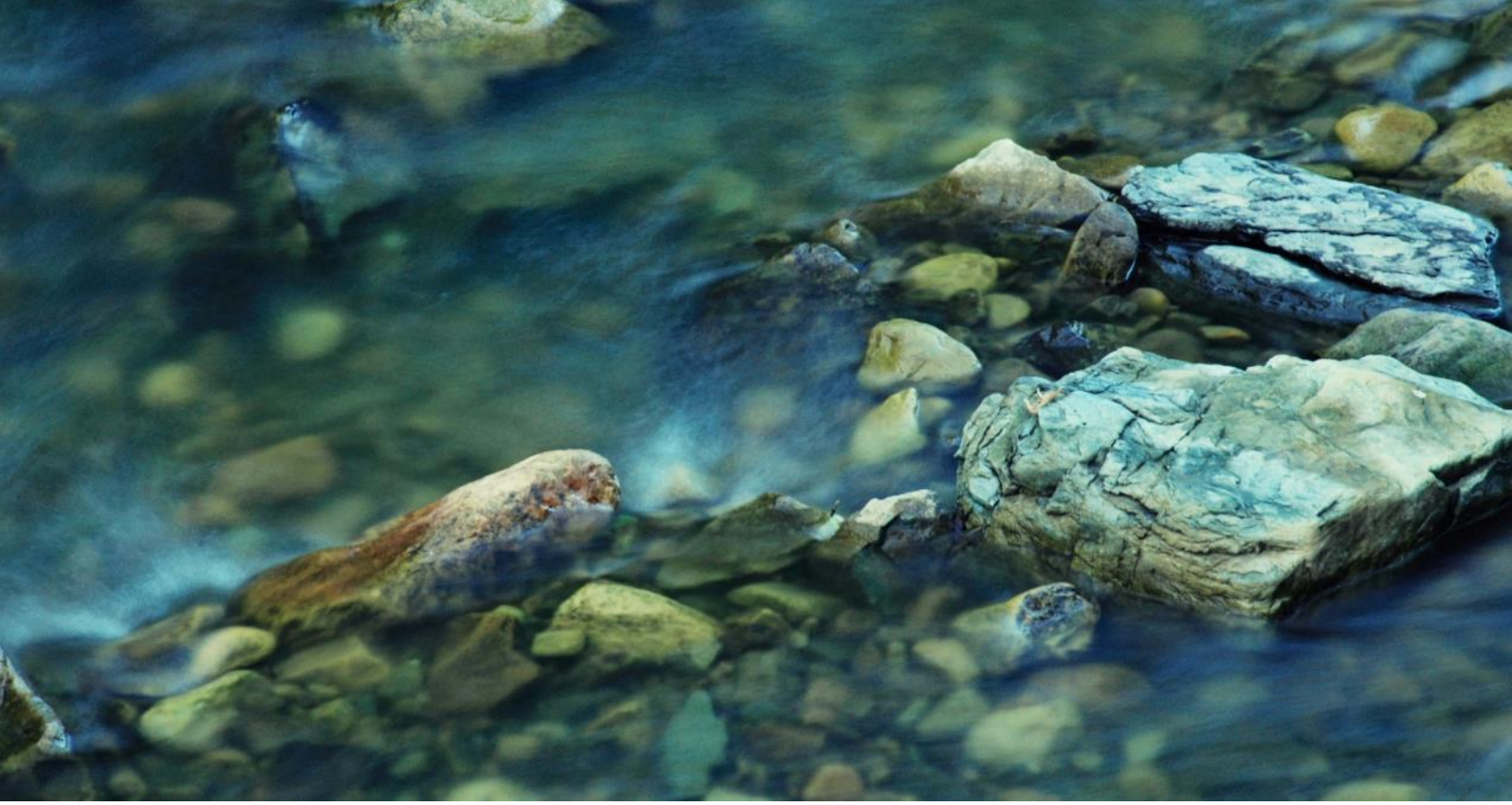

August 2019
Hudson River PCBs Superfund Site

ANCHOR QEA

2017 Water and Fish Data Summary Report

Prepared for General Electric Company

606421

August 2019
Hudson River PCBs Superfund Site

2017 Water and Fish Data Summary Report

Prepared for
General Electric Company
Schenectady, New York

Prepared by
Anchor QEA, LLC
Saratoga Springs, New York

In conjunction with
Environmental Standards, Inc.
Valley Forge, Pennsylvania

Project Number: 160469-04.01

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ABBREVIATIONS

ASTM	ASTM International
BMP	Baseline Monitoring Program
CAM	corrective action memorandum
cfs	cubic feet per second
COC	chain-of-custody
DO	dissolved oxygen
DQO	data quality objective
DSR	Data Summary Report
EDD	electronic data deliverable
EDI	equal discharge increment
EDV	electronic data verification
EPA	U.S. Environmental Protection Agency
ESI	Environmental Standards, Inc.
g	gram
GE	General Electric Company
L	Liter
LCS	laboratory control spike
LD	laboratory duplicate
m	meter
MDL	method detection limit
mg/kg	milligrams per kilogram
mg/L	milligrams per liter
mm	millimeter
MS	matrix spike
MSD	matrix spike duplicate
ND	non-detect
ng/L	nanograms per liter
NYSDEC	New York State Department of Environmental Conservation
OMM	operation, maintenance, and monitoring
Pace	Pace Analytical Services, Inc.
PCB	polychlorinated biphenyl
Phase 2 OMM Scope	*Operation, Maintenance, and Monitoring Scope for Phase 2 of the Remedial Action*
Phase 2 RAM QAPP	*Phase 2 Remedial Action Monitoring Quality Assurance Project Plan*
QA	quality assurance
QA/QC	quality assurance/quality control

QC	quality control
RA	Remedial Action
RAMP	Remedial Action Monitoring Program
RL	reporting limit
RPD	relative percent difference
SM	standard method
SOP	Standard Operating Procedure
SOW	*Statement of Work for Remedial Action and Operation, Maintenance, and Monitoring*
TSS	total suspended solids

1 Introduction

This 2017 Water and Fish Data Summary Report (DSR) has been prepared on behalf of the General Electric Company (GE) by Anchor QEA, LLC, in conjunction with Environmental Standards, Inc. (ESI), to document water and fish data collected from the Hudson River in 2017.

The sampling conducted in 2017 constituted the first full year of sampling following GE's completion of the Remedial Action (RA) in the Upper Hudson River in 2016. The *Statement of Work for Remedial Action and Operation, Maintenance, and Monitoring* (SOW) issued by the U.S. Environmental Protection Agency (EPA) in 2010 under the Consent Decree for the Hudson River PCBs Superfund Site requires GE to conduct a long-term operation, maintenance, and monitoring (OMM) program following the completion of the RA. The overall scope of the OMM program was set forth in the *Operation, Maintenance, and Monitoring Scope for Phase 2 of the Remedial Action* (Phase 2 OMM Scope), which is Attachment E to the SOW. The post-remediation OMM activities described in the Phase 2 OMM Scope include water column, fish, and sediment monitoring to assess long-term recovery. The long-term water column, fish, and sediment monitoring activities to be conducted as part of the OMM program will be described in *a Long-Term Operation, Maintenance, and Monitoring Plan for Water, Fish, and Sediment Monitoring*, to be submitted at a later date. Therefore, with EPA's agreement, the sampling programs conducted in 2017 and detailed in this DSR were conducted in accordance with GE's approved *Phase 2 Remedial Action Monitoring Quality Assurance Project Plan* (Phase 2 RAM QAPP; Anchor QEA and ESI 2012).

This DSR includes a summary of the surface water and fish sampling programs conducted by GE in the Hudson River in 2017. The surface water monitoring included the collection and analysis of water samples and collection of water quality data at Upper and Lower Hudson River sampling locations. Because the water samples collected in 2017 were collected in accordance with the Phase 2 RAM QAPP, they were categorized as off-season samples. The fish sampling program included the collection and analysis of fish samples from locations in the Upper and Lower Hudson River.

The objectives of this DSR are to describe the methods, summarize the data, and present the results of the applicable data quality assessments associated with the 2017 water and fish sampling and analysis activities. Data interpretation presented in this report is limited to assessments of data quality and usability.

2 Methods

2.1 Water Sampling Program

The 2017 water monitoring program included the collection of samples within remediated areas as well as from background stations located upstream of the project area and locations in the Lower Hudson River (Figures 2-1a through 2-1g). The stations monitored in 2017 are located at or near stations historically used for the Hudson River Baseline Monitoring Program (BMP) sampling (QEA and ESI 2004) and the Remedial Action Monitoring Program (RAMP). The general locations of these stations (from upstream to downstream) are as follows:

- Bakers Falls (background station)
- Rogers Island (background station)
- Thompson Island (project area monitoring)
- Schuylerville (project area monitoring)
- Waterford (project area monitoring)
- Albany (Lower River monitoring)
- Poughkeepsie (Lower River monitoring)

A summary of the water monitoring station locations and the 2017 sampling schedule is presented in Table 2-1 and summarized below.

Sampling was conducted at the Bakers Falls and Rogers Island stations monthly from May through November. The Rogers Island station is only accessible by boat; therefore, it was not sampled during the winter or during high-flow periods. The Bakers Falls station was sampled only when samples were collected from the Rogers Island station.

The Thompson Island and Schuylerville manual sampling locations were sampled weekly from May 1 to December 8, 2017. These stations are only accessible by boat and, therefore, are not sampled during the winter or during high-flow periods.

The Waterford manual sampling location was sampled weekly from February 23 through December 28, 2017. Ice in the Hudson River prevented sampling during the first part of 2017. Samples were collected from the Route 4 bridge or by boat, depending on flow and weather conditions.

The Lower Hudson River stations at Albany and Poughkeepsie were sampled monthly from May through November 2017. These stations are only accessible by boat and, therefore, were not sampled during the winter.

2.1.1 Sample Collection Procedures

Water monitoring in 2017 included the manual collection of samples at stations along the river from a boat or from a bridge, depending on river flow and seasonal conditions. Water sampling was conducted following procedures detailed in the Phase 2 RAM QAPP and summarized in this section.

Sampling was performed during 2017 at Bakers Falls, Rogers Island, Thompson Island, Schuylerville, Waterford, Albany, and Poughkeepsie. At all stations except Rogers Island, a custom-designed, multiple-aliquot depth-integrating sampler was lowered through the water column to collect depth-integrated samples. At Rogers Island, due to the shallow water depth at this location, surface grab samples were collected by immersing sample containers directly into the river.

At Bakers Falls, depth-integrated samples were taken at the approximate centroid of the river cross-section from the downstream side of the Bakers Falls Bridge (County Route 27 Bridge). At Rogers Island, surface grab samples were collected at a point near the center of the channel, upstream of all dredge areas but downstream of the former Fort Edward Dam. Depth-integrated composite samples were collected in the river by boat at the Thompson Island, Schuylerville, and Waterford locations used during the BMP (QEA and ESI 2004). Samples were composited from six equal discharge increment (EDI) locations across the river at Thompson Island and Schuylerville and from five EDI locations at Waterford (Figures 2-1c to 2-1e). Samples were collected downstream of the southern end of Thompson Island. At Schuylerville, samples were collected upstream of the State Route 29 Bridge at two EDI locations on the west side of Schuyler Island and four EDI locations on the east side. At Waterford, samples were collected along the upstream side of the State Route 4 Bridge. When the river at Waterford was not accessible by boat (e.g., during inclement winter weather), samples were collected from the bridge at five EDI locations whenever the river was ice free.

Lower Hudson River sampling at Albany and Poughkeepsie consisted of collecting a single depth-integrated composite sample by boat from each station at the approximate centroid of the river.

2.1.2 High-Flow Sampling

Section 2.5.3.2 of the Phase 2 RAM QAPP required that high-flow monitoring be performed at Waterford when river flow exceeds 15,000 cubic feet per second (cfs) at Fort Edward or 22,500 cfs at Waterford. At EPA's request, a second high-flow sampling location was added at the Dix Bridge in Schuylerville. The Dix Bridge is located immediately to the east of Lock 5 in Schuylerville, approximately 1.5 miles north of the routine weekly Schuylerville in-river location at the Route 29 Bridge (Figures 2-1a to 2-f). The Dix Bridge location was selected due to safety concerns associated with accessing the river from the Route 29 Bridge.

The Phase 2 RAM QAPP provides that the timing of the high-flow sample collection will be based on instantaneous flow obtained at the United States Geological Survey's Fort Edward gaging station. Thus, high-flow sampling was generally initiated based on the Fort Edward high-flow criteria. However, when flows were elevated at Waterford, but not at Fort Edward, due to localized precipitation events, the Waterford gage and other upstream tributary gages were used to determine the sampling schedule.

The river flow exceeded the specified criteria for Fort Edward and Waterford on numerous occasions in 2017. Six high-flow events were sampled at Dix Bridge and at Waterford (Section 4.1.2).

2.1.3 Analytical Program

The 2017 water analytical program is summarized in Table 2-2. The analytical method for polychlorinated biphenyls (PCBs) was changed in 2017 to EPA Method 1668C due to the facts that the historical project laboratory, operated by Pace Analytical Services, LLC, in Schenectady, New York (Pace Schenectady), which had analyzed water samples by the modified Green Bay Method (mGBM), had closed and that the mGBM was not offered at any other commercial laboratory facility. The specific analytical methods used are described in detail in Corrective Action Memorandum (CAM) No. 14 (May 17, 2017), which is provided in Appendix A.

Vista Analytical Laboratory of El Dorado Hills, California, performed PCB analysis of water samples using EPA Method 1668C, as described in Standard Operating Procedure (SOP) 31 (Attachment 4 to CAM No. 14). Pace Analytical Services, LLC, of Minneapolis, Minnesota (Pace Minneapolis), analyzed 1-Liter (L) water samples for total suspended solids (TSS) following the standard EPA protocol (Standard Method [SM] 2540D) for the analysis of suspended sediment, with modifications consistent with the ASTM International (ASTM) D3977-97 Standard Test Methods for Determining Sediment Concentration in Water Samples, Test Method B – Filtration (Attachment 2 to CAM No. 14).

2.1.4 Water Quality Field Parameters

Surface water quality measurements were taken at mid-depth in the water column at each EDI or single point location at the time of sample collection. The measurements were made using a multi-parameter water quality meter and included temperature, specific conductivity, pH, dissolved oxygen (DO), and turbidity.

2.2 Fish Sampling Program

The RAMP fish monitoring program continued in 2017 in accordance with the Phase 2 RAM QAPP. Adult fish were sampled in the spring, and yearling pumpkinseed and forage fish were sampled in

the fall. Fish collection was performed along transects at stations within four pools (also referred to as reaches) of the Upper Hudson River and at three stations in the Lower Hudson River, as follows:

- Feeder Dam Pool – Reference Area (one station)
- Thompson Island Pool (five stations)
- Northumberland Pool (four stations)
- Stillwater Pool (five stations)
- Albany/Troy (one station – below Federal Dam in spring; Albany between Dunn Memorial Bridge and Route 90 Bridge in fall)
- Catskill (one station; spring only)
- Tappan Zee (one station; spring only)

The spring and fall fish sampling transect locations are depicted in Figures 2-2a-k and 2-3a-i, respectively.

2.2.1 Spring Sampling

Spring fish sampling occurred on April 18 through 20, May 18 and 19, June 12 through 16, and June 19 and 20, 2017 (Table 2-3). During sampling, adult species of black bass (smallmouth bass [*Micropterus dolomieu*] and largemouth bass [*M. salmoides*]), yellow perch [*Perca flavescens*], and ictalurids (brown bullhead [*Ameiurus nebulosus*] and yellow bullhead [*A. natalis*]) were targeted from the 15 stations in the Upper Hudson River. The Lower Hudson River stations were sampled for smallmouth bass (Albany/Troy), channel catfish (*Ictalurus punctatus*; Albany/Troy), brown bullhead (Catskill), perch (yellow perch and white perch [*Morone americana*]; Albany/Troy and Catskill), and striped bass (*M. saxatilis*; Albany/Troy, Catskill, and Tappan Zee stations). As directed by EPA, largemouth bass were not targeted at Albany/Troy (the only black bass collected were smallmouth bass), and no black bass were targeted at Catskill. Adult fish meeting the legal or edible total length criteria were targeted as follows:

- Black bass: 305 millimeters (mm) or larger
- Bullhead/catfish: 200 mm or larger
- Yellow perch: 170 mm or larger
- White perch: 160 mm or larger
- Striped bass: 457 mm or larger

Twenty fish samples from Thompson Island Pool arrived at the laboratory one week late due to an error by the shipping company. Those samples were out of range of the temperature standard and therefore were not submitted for analysis. As a result, those 20 fish samples were re-collected from the Thompson Island Pool. The re-collected fish samples were five black bass, two bullhead, and five yellow perch at TD1, five yellow perch at TD2, and three black bass at TD4.

A total of 495 fish samples were submitted for analysis from the spring sampling stations (Figures 2-2a through 2-2k): 125 black bass, 145 ictalurids, 145 perch, and 60 striped bass. The number and species of fish collected at each station during the spring 2017 sampling are shown in Table 2-3.

As indicated in Table 2-3, it was not possible to collect the full number of targeted fish at some of the stations within the Upper Hudson River pools (or reaches) due to limited availability. Consistent with the approach followed in previous years, additional fish were collected (if available) at adjacent stations (within the same reach), as described below, with the goal of providing a similar number of fish as in previous years. This approach has been followed to improve the comparability of the reach-wide average PCB concentrations from year to year.

The targeted number of individuals per station for each species was collected successfully in the Feeder Dam Pool, the Stillwater Pool, Albany/Troy, Catskill, and Tappan Zee.

In the Thompson Island Pool, 30 individuals per species were targeted: five individuals per species at stations TD1, TD2, TD3, and TD4, and 10 individuals per species at the historical New York State Department of Environmental Conservation (NYSDEC) station at TD5. Due to the limited availability of bullhead at TD1 (two fish) and the absence of bullhead at TD3, a total of eight extra bullhead were collected at TD2. Due to the absence of bullhead at TD 4, five extra bullhead were collected at TD5.

In Northumberland Pool, 25 individuals per species were targeted for collection: five individuals per species at stations ND1, ND2, and ND3, and 10 individuals per species at ND5 to make up for not sampling at ND4, which was abandoned after the first year of the BMP due to the lack of fish and habitat. Five individuals per species were collected at ND1 as well as five extra bullhead and two extra yellow perch to make up for the paucity of those species at ND2. Five bass and three yellow perch were collected at ND2. Five individuals per species were collected at ND3 and 10 individuals per species were collected at ND5.

2.2.2 Fall Sampling

Forage fish and yearling pumpkinseed were collected from August 29 through September 1, 2017, from the Upper Hudson River stations and the Albany/Troy station (Figures 2-3a through 2-3i). A total of 175 samples (125 yearling pumpkinseed and 50 forage fish composite samples) were collected from all stations (Table 2-4). Forage fish species collection targeted spottail shiner (*Notropis hudsonius*) or the following substitute species: fallfish (*Semotilus corporalis*), spotfin shiner (*Cyprinella spiloptera*), mimic shiner (*N. volucellus*), banded killifish (*Fundulus diaphanus*), golden shiner (*Notemigonus chrysoleucas*), or bluntnose minnow (*Pimephales notatus*) (one species per composite), based on availability. Multiple forage fish (whole body) of the same species were used to form composite samples. Composites typically consisted of similar-sized fish in multiples of five with a target mass of at least 20 grams (g) to provide adequate sample volume for laboratory analysis. A

total of 50 composites were targeted from the stations sampled in the fall (10 composites from each pool and the Albany/Troy station; Table 2-4). Yearling pumpkinseeds were captured from each pool and the Albany/Troy station and submitted as whole-body individual samples. Pumpkinseeds were considered yearlings if they were between 70 and 130 mm in total length, in accordance with the requirements in the Phase 2 RAM QAPP. The numbers of pumpkinseed and forage fish collected at each station in the fall sampling are shown in Table 2-4.

2.2.3 Sampling Methods

Electrofishing and angling were performed to collect target species. Electrofishing was accomplished with an 18-foot boat equipped with a variable-output, gas-powered direct-current generator. Operating amperage was adjusted according to water conductivity to minimize injury to fish; stunned fish were immediately removed from the electrical field using dip nets to minimize the duration of the shock. At the Tappan Zee location, striped bass were captured by angling from a chartered fishing vessel and as bycatch from the permitted bait net deployed by the charter captain. Higher water conductivity in this area limits electrofishing effectiveness. Also, as requested by NYSDEC, standard gill netting techniques, which include deployment of several nets for short durations throughout the sampling location, could not be used due to the possibility of bycatch of Atlantic sturgeon (*Acipenser oxyrinchus oxyrinchus*) or shortnose sturgeon (*A. brevirostrum*), which are on the federal Endangered Species List. Fish were held in live wells or buckets with frequent water changes until processed.

Sampling and handling methods were consistent with the SOP for Fish Collection and Processing (Phase 2 RAM QAPP, Appendix 3.5-1). Adult fish were collected along transects at each station during spring 2017. Spring transects were approximately 200 to 2,000 meters (m) in length and were located parallel to the shoreline in water approximately 1 to 3 m deep (Figures 2-2a through 2-2k). Fish were collected in fall 2017 generally along the same transects sampled in the spring. Transects at some stations were modified based on historical NYSDEC yearling pumpkinseed sampling locations that were in slightly different areas from adult fish locations. Fall transects were approximately 200 to 1,000 m in length and were located parallel to the shoreline in water approximately 1 to 3 m deep (Figures 2-3a through 2-3i).

Fish were processed as soon as possible after collection. Just prior to processing, each fish was euthanized with a blow to the head. For each specimen, the date of collection, a unique ID number or code, the station location (including coordinates), species, total length in millimeters (to nearest millimeter), weight in grams (to nearest 1.0 g for adult fish, 10 g for striped bass, and 0.1 g for yearling pumpkinseed and forage fish), and method of collection were recorded in the RAMP fish field database. Measurements were made with calibrated instruments. Each sample was wrapped in clean aluminum foil (shiny side out), labeled, placed in a plastic resealable storage bag, and kept on ice following data processing. The same information was also collected for composited fish, including

number of individuals within the composite. Visually identified external abnormalities were noted in the database.

Chain-of-custody (COC) forms were generated after data were entered into the database and samples were grouped by species and location for each COC form. Samples were kept on ice and shipped for overnight delivery to Pace Analytical Services, LLC, of Green Bay, Wisconsin (Pace Green Bay), for analysis.

2.2.4 Laboratory Sample Preparation

The project laboratory for fish analysis was changed to Pace Green Bay in 2017 due to the closure of the historical project laboratory, Pace Schenectady, that was previously used to analyze fish samples. The analytical methods used are described in detail in CAM No. 15 (August 15, 2017; revised February 6, 2018), which is provided in Appendix A. Pace Green Bay SOPs were directly based on the prior Pace Schenectady SOPs with the modifications detailed in CAM No. 15.

Fish preparation (scaling, skin removal from ictalurids, filleting, and sex determination) was conducted by Pace Green Bay following protocols described in Pace SOP S-GB-L-009-REV.01 (Attachment 1 to CAM No. 15). Striped bass, black bass, perch, and ictalurids were prepared by removing the left fillet, including the rib cage, for analysis; the right fillet was only included if needed for sufficient sample mass. Fish tissue, either whole body or fillet, was homogenized following the methods outlined in Pace SOP S-GB-L-009-REV.01. Extraction of fish tissue was accomplished via Pace SOP S-GB-O-068-REV.01 (Attachment 2 to CAM No. 15).

2.2.5 Analytical Program

Fish samples were analyzed by Pace Green Bay for Total Aroclor PCBs according to a modification of Method SW-846 8082A, as described in CAM No. 15 (Pace SOP S-GB-O-067-REV.01; Attachment 3 to CAM No. 15). Additionally, fish samples were analyzed by Pace Green Bay to determine lipid content according to the methods outlined in Pace SOP S-GB-O-068-REV.01 (Attachment 2 to CAM No. 15).

2.3 Quality Assurance Project Plan Updates and Technical Memoranda

As noted above, two CAMs were developed to describe the analytical methods used for the 2017 water and fish samples: Updates to the Analytical Program Approach and Procedures for water analysis (CAM No. 14, dated May 17, 2017) approved by EPA on July 11, 2017; and Updates to the Analytical Program Approach and Procedures for fish preparation and analysis (CAM No. 15, dated August 15, 2017; revised February 6, 2018) approved by EPA on January 26, 2018. As also noted, those CAMs and EPA approvals are provided in Appendix A.

In addition, two technical memoranda were developed to describe certain data quality issues that were identified in the 2011 data and document the resolution of those issues. These memoranda are also included in Appendix A and consist of the following:

- Pace GB 2017 Fish Matrix Spike Evaluation (Technical Memorandum dated August 16, 2019)
- Results of Re-Extraction of Sample OWS-THIS-T170726143612 (Technical Memorandum dated August 19, 2019)

The first of these memoranda addresses a quality assurance/quality control (QA/QC) deviation relating to the quantitation of Aroclor 1242. The second discusses the reanalysis of a sample that exhibited PCB concentrations higher than typically observed at Thompson Island.

3 Quality Assurance/Quality Control

3.1 Field Quality Assurance/Quality Control

The procedures followed for QA/QC sample collection during the 2017 sampling program were consistent with those specified in the Phase 2 RAM QAPP unless noted otherwise. Program components that were consistent with the Phase 2 RAM QAPP included the following:

- Sample handling and custody requirements
- QC requirements
- Precision, accuracy, representativeness, comparability, completeness, and sensitivity
- Inspection/acceptance requirements for supplies and consumables
- Data management
- Data validation
- Performance audits
- Corrective action
- Verification and validation methods

Field QA/QC samples were prepared to allow for evaluation of data quality. Field QA/QC samples included equipment blanks, matrix spike/matrix spike duplicate (MS/MSD), and field duplicate samples. Field QA/QC samples for water column samples included equipment blank samples, blind duplicate samples, and laboratory duplicate (LD) samples. Fish sampling field QA/QC samples were generated in the laboratory because fish sampling does not include the collection of field QA/QC samples as part of the study design.

3.1.1 Water Sampling Instrument Calibration

Water quality measurements for temperature, specific conductivity, pH, DO, and turbidity were made during sample collection using a YSI 6920 water quality meter equipped with multi-parameter probes. The probes were calibrated in accordance with the user manual prior to use. The calibration records of the multi-parameter probes were recorded in a calibration log as specified in Section 2.3.7 of the Phase 2 RAM QAPP.

3.1.2 Fish Sampling Instrument Calibration

Balances used to weigh fish were checked with calibration weights each day prior to sampling. Calibration checks were recorded in a field log. A YSI 6920 water quality meter was used at each station. This meter was calibrated prior to spring and fall sample collection in accordance with the user manual. Equipment was maintained and repaired in accordance with the manufacturer's specifications (Section 3.6 of the Phase 2 RAM QAPP). Prior to use, each major piece of equipment

was cleaned, decontaminated, checked for damage, and repaired if needed. Field calibration activities were noted in a field log notebook.

3.1.3 Equipment Blanks

Equipment blanks were collected for PCB analysis at a rate of 1 per 20 samples. These samples were collected approximately monthly throughout the season. Equipment blanks were not applicable to the TSS analysis. Equipment blanks for water sampling were collected using a representative clean, individual subsample collection container in accordance with the water column sample collection SOPs (Appendices 2.3-1, 2.3-2, and 2.4-1 of the Phase 2 RAM QAPP).

Equipment blanks for fish tissue samples were not required in the approved Phase 2 RAM QAPP.

3.1.4 Field Duplicates

Sample duplicates were collected in the field (co-located with the environmental sample) following sampling procedures detailed in the water column sample collection SOPs (Appendices 2.3-1 and 2.4-1 of the Phase 2 RAM QAPP). These samples were submitted to the analytical laboratory "blind" without any indication of the actual sample location. Field duplicates were generally prepared at a rate of 5% or greater of the total number of environmental samples (at least 1 duplicate sample per 20 samples) as specified in the Phase 2 RAM QAPP. Because it is impossible to collect field duplicates of fish, laboratory duplicates (LDs) for fish were generated in the laboratory by splitting the homogenate.

3.1.5 Laboratory Duplicates/Matrix Spikes

The water program included analysis of LD samples for TSS at a rate of one per sample batch (up to 20 samples). Some of the sample batches for TSS did not include the required LD, but an overall rate of 5% was met.

MS/LD samples were analyzed at a minimum rate of one pair per sample batch (up to 20 samples) for fish samples.

3.2 Laboratory Quality Assurance/Quality Control

Laboratory QA/QC was conducted on samples submitted to and analyzed by GE's contract laboratories.

3.2.1 Method Blanks

Method blanks were prepared and analyzed by the laboratory at a rate of at least one per analytical batch. Method blanks for water samples consisted of laboratory-prepared blank water that was processed along with the batch of environmental samples, including all treatments performed on

actual samples. Method blanks for fish consisted of sodium sulfate that was processed along with the batch of environmental samples, including all treatments performed on actual samples.

3.2.2 Laboratory Control Spikes

Laboratory control spikes (LCSs) were analyzed at the rate of one per sample batch (up to 20 samples). LCSs consisted of laboratory-fortified method blanks. The purpose of analyzing LCSs is to demonstrate the accuracy of the analytical method.

3.2.3 Temperature Blanks

A temperature blank was provided in each cooler sent from the field to the laboratory. The purpose of this sample was to document the temperature of the cooler upon arrival at the laboratory.

3.3 U.S. Environmental Protection Agency Split Samples

EPA did not collect split water or fish samples during 2017.

3.4 Field and Laboratory Audits

Field audits of the water column collection activities performed by Anchor QEA field personnel were conducted by ESI on June 15 and November 1, 2017. Field audits of 2017 spring and fall fish collection activities performed by Anchor QEA field personnel were conducted by ESI on May 9 and June 14, 2017, and on August 28, 2017, respectively. These audits were conducted as described in Section 11.1.2 of the Phase 2 RAM QAPP. The field audits indicated that the field crews conducted their work in a professional manner and complied with the procedures outlined in the Phase 2 RAM QAPP and applicable SOPs. The field audits also indicated that consistent sample collection and processing procedures were used during 2017. A few minor issues identified during the audits are discussed in the audit reports (provided in Appendix B). The issues identified in the audit reports did not jeopardize the data quality objectives (DQOs) of the project. When possible, the recommendations were discussed with the field team at the time of occurrence. A debriefing meeting was held with Anchor QEA field personnel at the conclusion of each audit. The field crews incorporated recommendations, as appropriate.

Laboratory audits were conducted by ESI personnel on November 14, 2017, for Vista Analytical Laboratory (with respect to PCB analyses for water samples) and from February 13 to February 15, 2018, and April 19 to April 20, 2018, for Pace Green Bay (with respect to PCB and lipids analyses for spring and fall fish samples). An audit was not conducted for Pace Minneapolis, which only performed TSS analyses for water samples. The audits were conducted as described in Section 11.2.3 of the Phase 2 RAM QAPP and intended to provide feedback on laboratory operating issues with respect to method compliance, laboratory systems, and good laboratory practices.

The audit reports for the contract laboratories are also included in Appendix B. The audits found that the laboratories were adhering to the project-specific methods and QA requirements.

3.5 Data Management

Data collected under the water and fish sampling programs have been stored in an electronic database. Specialized application modules, outlined in Sections 3.6.1 through 3.6.2, were used to automate data collection, data evaluation, and data integration.

3.5.1 Field Sample Data Collection System

The water monitoring program collected field data, including sample collection information and water quality data obtained with instruments, and water samples for laboratory analysis. Field data were entered electronically in a field database application designed to support the monitoring program. The field database application comprised electronic data entry forms and data export functions designed to support efficient workflow and accurate data recording. Features included data entry fields with valid value selection lists to limit entry errors and automated data generation for field values based on user-entered information to limit transcription errors. Functions also included sample label and COC form generation capabilities for samples that were sent to laboratories for analysis. Further, these applications had procedures for electronic data deliverable (EDD) generation from field databases to facilitate accurate data import into the central project database.

Water quality field parameters were measured at manually sampled locations. These field measurements were entered into the field application and uploaded through a field-based EDD and automatically checked for valid values before being stored in the project database. If any data did not pass these checks, an error log was generated for review by designated data QC personnel.

For the fish sampling program, field-generated data were entered into a field database via custom-designed forms developed in Microsoft Access. This custom application facilitated data entry and management of the collected field data for the project by capturing, managing, and maintaining field data, including sample ID creation, field and fish sample observations, electronic COC creation, and sample label creation. These forms limited the possibility of data entry or transcription errors by including valid value selection lists for certain required fields. In addition, several data fields were populated automatically to further reduce data entry and transcription errors.

3.5.2 Laboratory Data Checker

Stage 2a EDDs submitted by the analytical laboratories to the data management system were automatically checked for data reliability according to various criteria, including valid values, data types, and format, as described in the Phase 2 RAM QAPP. If errors were detected, the laboratory was notified and the file corrected by the laboratory for loading into the data management system.

3.5.3 Data Verification Module

An automated data verification module verified analytical data submitted by the laboratory, reviewed data against the performance specifications provided for the project based on field and laboratory QC samples, produced exception reports, and loaded qualified results to the RAMP database.

Automated electronic data verification (EDV) was performed on 100% of the analytical results received using the batch QC results provided by the laboratories in the EDDs. The term "verification" indicates that a criteria-based data quality evaluation was performed to assess sample and method completeness and that laboratory QC results met limits defined in the Phase 2 RAM QAPP. These checks, following EPA Stage 2a validation protocols, were used to qualify the data, as necessary. The following specific measures were evaluated during verification and by the associated criteria and are discussed in the Phase 2 RAM QAPP:

- Holding times
- Accuracy (by evaluating LCS and MS/MSD recoveries)
- Precision (by evaluating LD results)
- Field duplicate sample precision
- Blank contamination (laboratory method blanks and field generated blanks)
- Surrogate compound recoveries
- Percent solids

3.6 Data Validation

EDV and data validation (where necessary) were conducted after samples were collected and analyzed. The usability of the analytical data was assessed using a tiered approach. All data initially underwent an EDV, which provided the first test of the quality of the results. This automated process assessed data usability by evaluating completeness and laboratory batch QC results, as described in Section 3.5.3.

Full validation (i.e., manual qualitative and quantitative checking) included an evaluation of documented QA/QC measures through a review of tabulated QC summary forms and raw instrument data. The validation results were also compared to the results of the electronic verification for the same set of data, which provided an indication of the accuracy of the electronic verification process. Verification and validation findings are discussed in Section 5.1.

3.6.1 Water Data Validation

Full data validation was performed on a minimum of 5% of the PCB and TSS data from water samples, as presented in Table 3-1. The overall percentage of data validated for the Phase 2 RAMP data included in this DSR for each analytical technique is presented in Table 3-1.

3.6.2 Fish Data Validation

Full data validation was performed on a minimum of 5% of the PCB data (for Aroclor PCBs) from fish tissue samples, as presented in Table 3-1 Data generated from both the spring and fall fish collections were selected for validation to identify potential issues.

3.7 Sample Archives

The 2017 RAMP water sample extracts for PCB analysis and homogenized tissue from fish samples were required to be archived until the holding time was exceeded (frozen below -10°C for extracts and below -18°C for fish tissue), as specified in Section 10.1.3 of the Phase 2 RAM QAPP as follows:

Sample/PCB Extract Matrix	Archive Time
Water Sample Extract	Until holding time is exceeded
Homogenized Fish Tissue	1 year from collection
Fish Tissue Extract	1 year from collection

GE will provide EPA with the option of obtaining from GE some or all of the 2017 archived sample extracts and homogenized fish tissue samples. To the extent that EPA does not want those extracts or samples, GE will dispose of them.

4 Results

4.1 Water Sampling Results

4.1.1 Routine Monitoring Results

4.1.1.1 Polychlorinated Biphenyls

A total of 136 routine samples (129 environmental samples plus 7 duplicate samples) (also referred to as off-season samples) were analyzed for PCBs by Method 1668 in 2017. PCB results ranged from 0.27 nanograms per liter (ng/L) to 46.5 ng/L. Summary statistics by station are presented in Table 4-1, and the data are included in the database provided in Appendix C.[1]

4.1.1.2 Total Suspended Solids

A total of 135 samples (129 environmental samples plus 6 duplicate samples) were analyzed for TSS. Results ranged from non-detect (ND) to 74.8 milligrams per liter (mg/L). Summary statistics are presented in Table 4-1, and the data are included in Appendix C.

4.1.2 High-Flow Monitoring Results

High-flow sampling was conducted during six events in 2017: on February 26 through March 3, April 4 through April 13, April 23 through April 24, May 7 through May 15, June 7, and July 2 through July 3. Manual samples were collected at the Dix Bridge in Schuylerville and the State Route 4 Bridge in Waterford. Samples were analyzed for PCBs by Method 1668 and for TSS.

4.1.2.1 Polychlorinated Biphenyls

Fifty high-flow samples (47 environmental samples plus 3 duplicates) were analyzed for PCBs by Method 1668. Results ranged from 3.0 to 37.7 ng/L. Summary statistics are presented in Table 4-1, and the data are included in Appendix C.

4.1.2.2 Total Suspended Solids

Fifty high-flow samples (47 environmental samples plus 3 duplicates) were analyzed for TSS. Results ranged from 4.4 to 283 mg/L. Summary statistics are presented in Table 4-1, and the data are included in Appendix C.

[1] The summary results presented in Table 4-1, for both PCBs and TSS and for both routine and high-flow monitoring, are for detected results only and samples with duplicates are reported as the average of the parent and duplicate sample results. By contrast, the results presented in this section consist of the range of all sample results with duplicates reported independently from parent samples. Due to these differences, some of the minimum and maximum concentrations reported in this section do not match those reported in Table 4-1.

4.1.3 Water Quality Results

Summary statistics for general water quality parameters, including DO, turbidity, pH, specific conductance, and water temperature, are presented in Table 4-2. Water quality data are included in Appendix C.

4.2 Fish Sampling Results

4.2.1 Polychlorinated Biphenyls

A total of 650 fish samples were collected from the Hudson River during the 2017 field sampling season (475 samples in spring, 175 samples in fall). All 650 samples were submitted for Aroclor PCB analysis using Method SW846 8082 (S-GB-O-067-REV.01).

The 2017 PCB results are included in the fish dataset presented in the RAMP fish database (Appendix D).

4.2.1.1 Black Bass

Aroclor PCBs were detected in 119 of 125 black bass samples (including largemouth and smallmouth bass) (Table 4-3, Figure 4-1).

4.2.1.2 Ictalurids

Aroclor PCBs were detected in 141 of 145 ictalurid samples (brown bullhead, yellow bullhead, and channel catfish) (Table 4-4, Figure 4-2).

4.2.1.3 Perch

Aroclor PCBs were detected in 136 of 145 perch samples (including yellow perch and white perch) (Table 4-5, Figure 4-3).

4.2.1.4 Striped Bass

Aroclor PCBs were detected in 60 of 60 samples of striped bass (Table 4-6, Figure 4-4).

4.2.1.5 Pumpkinseed

Aroclor PCBs were detected in 125 of 125 pumpkinseed samples (Table 4-7, Figure 4-5).

4.2.1.6 Forage Fish

Aroclor PCBs were detected in 50 of 50 composite samples of forage fish (spottail shiner, golden shiner, fallfish, spotfin shiner, and bluntnose minnow) (Table 4-8, Figure 4-6).

4.2.2 Lipids

Summary statistics are included in Tables 4-9 through 4-14 for each species by Hudson River pool and Lower Hudson River station. The lipid results are included in the fish dataset presented in the RAMP fish database (Appendix D).

4.2.3 Sex

Results for fish sex are presented in this section by species. Summary statistics are included in Table 4-15 for each species by Hudson River pool. A total of 650 fish were collected for analysis during the 2017 field sampling season (475 in spring, 175 in fall). The sex of each individual fish collected in spring was determined and the results included in the fish dataset presented in the 2017 RAMP fish database (Appendix D).

4.2.3.1 Black Bass

Fish sex was determined in all 125 of the black bass (largemouth bass and smallmouth bass); there were 73 males and 52 females (Table 4-15).

4.2.3.2 Ictalurids

Fish sex was determined in 134 of 145 ictalurids (brown bullhead, yellow bullhead, and channel catfish); there were 47 males and 87 females (Table 4-15).

4.2.3.3 Perch

Fish sex was determined in 118 of 145 perch; there were 106 males and 12 females (Table 4-15).

4.2.3.4 Striped Bass

Fish sex was determined in 59 of 60 striped bass samples analyzed from the Lower Hudson River stations; there were 30 males and 29 females (Table 4-15). Attempts to collect an approximately even number of males and females from Albany/Troy were made during collection by gently squeezing the fish along the flanks to see if eggs or milt were extruded; as noted, this effort was successful. The Albany/Troy station was sampled at the beginning of the Upper Hudson River sampling event, and the sex of 8 males and 12 females was confirmed in the laboratory (Table 4-15).

4.2.4 Fish Field Observations

Fish condition was assessed by field measurements and visual observation. External abnormalities were recorded to assess fish condition. Of the species examined, ictalurids appeared to have the most external abnormalities. The abnormalities observed are summarized by location in the following subsections.

4.2.4.1 Feeder Dam Pool (Reference Area)

Each of the smallmouth bass had blackspot. Several yellow perch had yellow grubs. Three yellow perch had black spot, and one had an attached leech. Three ictalurids had a lesion. Two ictalurids were missing barbels, and one had melanoma. Four of the pumpkinseed had split or eroded fins.

4.2.4.2 Thompson Island Pool

Most smallmouth bass had black spot, and four had eroded fins. One of the largemouth bass had black spot, and one had fin erosion. Many of the yellow perch had black spot and attached leeches; two had yellow grubs. Fin abnormalities, yellow grubs, abrasions, missing barbels, a damaged eye, a small cyst on the pectoral fin, and one lamprey wound were observed in the ictalurid group. Two pumpkinseeds had attached leeches, and two had black spot.

4.2.4.3 Northumberland/Fort Miller Pool

Black spot was observed in many smallmouth bass. Two smallmouth bass had fin erosion, and one had a leech attached. Several largemouth bass had fin erosion, and two had black spot. Many of the yellow perch exhibited eroded or damaged fins. Four yellow perch had black spot, and three had a leech attached. Several ictalurids had fin erosion and missing barbels. One ictalurid had a yellow grub, and one had a wound on the lower mandible. Three pumpkinseed had eroded caudal fins.

4.2.4.4 Stillwater Pool

Three smallmouth bass had black spot. Two smallmouth bass had fin erosion, and one had a yellow grub. Several largemouth bass had fin erosion. Three largemouth bass had black spot, and three had attached leeches. Several yellow perch had black spot and fin abnormalities such as erosion or deformed or split fins. Two yellow perch had attached leeches, and one had yellow grubs. Several ictalurids had fin erosion, and one had yellow grubs. Four pumpkinseeds had black spot.

4.2.4.5 Albany/Troy

Two smallmouth bass had eroded fins. One smallmouth bass had black spot, and one had a yellow grub on its anal fin. Two yellow perch were missing a piece of their upper caudal fin. Several ictalurids had missing barbels. One ictalurid had a wound on the dorsal side, and one had a lesion below the left eye. Three striped bass had a hook wound in the mouth.

4.2.4.6 Catskill

Three ictalurids had red spotting on the ventral side, and one had a lesion on the head. No abnormalities were observed in the striped bass.

4.2.4.7 Tappan Zee

One striped bass was observed to have fin erosion.

4.2.4.8 Condition Index

The weight and total length of captured fish were measured to assess fish condition. Condition index was determined using the following equation:

Equation 1

$$Condition\ Index(K) = \frac{Weight(g) * 100{,}000}{Length(mm)^3}$$

A condition index of 1.0 indicates a fish of normal condition. A condition index greater than 1.0 indicates a fish of better than average condition.

Except for the channel catfish at Albany/Troy, which had a condition index of 0.98, the black bass, ictalurids, perch, striped bass, and pumpkinseed captured during the 2017 RAMP fish sampling program had an average condition index greater than 1.0 (Figures 4-7 through 4-12). Forage fish captured during the 2017 RAMP fish sampling program had condition indexes ranging from 0.93 to 1.16 regardless of the station at which they were captured (Figure 4-12). Forage fish (fallfish) collected at TD5 were not used in the condition index calculation for that pool because length data collected for each individual fish were incorrectly transcribed to the field database.

4.3 Laboratory Analytical Data Packages

Electronic copies of the laboratory hardcopy data packages for water are included in Appendix E.

5 Data Quality

5.1 Validation/Verification

EDV and data validation of the analytical results were conducted as described in Sections 3.5.3 and 3.6 to provide an understanding of the analytical data quality. As discussed in Section 3.6, at least 5% of the 2017 PCB and TSS results from water samples and PCB and lipid results from fish tissue samples were subject to manual data validation. Appendix F lists, by constituent and method, each 2017 water and fish sample that was validated. Appendix G contains three data validation reports prepared for the 2017 sample data that were validated. These appendices provide the specific details of the data qualification resulting from the validation process.

Validation qualifier codes were placed next to the results in the project databases so that data users can quickly assess the qualitative or quantitative reliability of any result. The analytical database was then used to generate tabulated reports (data tables) of the validation results and qualifier codes. The final validated results are presented as data tables in the three data validation reports in Appendix G.

The same qualifier codes were used for both the data verification and validation processes, except where noted. The qualifier codes available for use and their definitions are as follows:

- Null: No qualifier code. The compound was detected and should be considered quantitatively and qualitatively valid based on the QC review.
- U: The compound/analyte was analyzed for but was not detected above the sample detection limit (reported as ND).
- UM: The compound/analyte was analyzed for but did not meet all the qualitative criteria of EPA Method 1668C and was not detected above the Estimated Maximum Possible Concentration (reported as ND).
- U* (fish) or UB (water): This compound/analyte should be considered ND because it was detected in a blank at a similar level.
- J: Quantitation is approximate (estimated) due to limitations identified during the QA review (or data validation).
- N: The analysis indicates that there is presumptive evidence to make a “tentative identification” of this compound/analyte.
- R: Unusable (rejected) result. The compound/analyte may or may not be present in this sample.
- UR: Unusable ND result. The compound may or may not be present in this sample.

- UJ: This compound/analyte was not detected, but the quantitation/detection limit is probably higher than reported due to a low bias identified during the QC review (reported as ND with a J qualifier).

The validation qualifier code field of the project database was queried to provide a tabulation of the number of results for each analysis fraction that were valid as reported (unqualified results and ND results U and, for PCBs only, UM), and those that were qualified during verification and validation with the qualifier codes identified above.

The percent usable and unusable data and the percent completeness were calculated for each analysis fraction according to the following equations:

Equation 2

% Usable data = Unqualified positive results + #U (+#UM for PCBs) + #U*/UB + #J + #UJ/total number of results

Equation 3

% Unusable data = #R + #UR/total number of results

Equation 4

% Completeness = Valid data as reported [unqualified positive results + #U (+#UM for PCBs)]/[total number of results – positive results <RL]

The percent completeness calculation does not include results qualified as estimated values (J) because they are below the sample-specific reporting limit (RL) but above the method detection limit (MDL). These results are not included in the completeness calculation because they are estimated values pursuant to standard EPA analytical data reporting conventions.

A summary of the data quality for the individual analytical fractions is presented in the following sections. The data quality has been described based on the percent completeness and percent usable results as follows:

Qualitative Data Quality	Percent Completeness	Percent Usable
Excellent	95%	100%
Very Good	85%	95%
Good	75%	90%
Above Average	65%	85%
Average	45%	80%
Poor	<45%	<80%

The percent completeness goal stated in the Phase 2 RAM QAPP is 95%. The Qualitative Data Quality index presented on this page was based on professional judgment and experience. It was developed to provide a qualitative framework to discuss the data quality. Although the description of data quality is based on criteria for both the percent completeness and percent usable data calculations, the percent usable data calculation is a more critical reflection of the data quality than the percent completeness calculation. Percent completeness reflects the percentage of the data that satisfied the DQOs (i.e., the percentage of unqualified data), whereas percent usability reflects the percentage of the data that have some qualitative or quantitative use, which is inclusive of the data that satisfied all the DQOs. The results of the percent completeness calculation do not indicate the nature of the qualification of the "incomplete" data. The data that are usable but qualitatively or quantitatively qualified may have no impact on the end use of the data, depending on what decisions need to be made based on those data. In other words, data that have low percent completeness may still be "100% usable" for decision-making purposes.

The following example calculations are provided for the percent completeness, percent unusable, and percent usable results presented in Table 5-1 for analyses of PCBs in water and the explanations in Table 5-1, Notes 6, 7, and 8, as follows:

1. Percent completeness is the sum of results that were valid as reported [unqualified positive results + U (+ UM for PCBs)]/[total number of results – positive results <RL].
   Example: 97.8% = [(14,591 + 8,545 + 1,767)/(31,504- 6,034)]*100
2. Percent unusable data is the sum of the results qualified R + UR/total number of results.
   Example: 0.0% = [(0 + 0)/31,504]*100
3. Percent usable data is the sum of the unqualified positive results + U [+ UM for PCBs] + UB + J + JN + UJ/total number of results.
   Example: 100% = [(14,591 + 8,545 + 1,767+ 93 + 6,506 + 0 + 2)/31,504]*100

5.1.1 Data Verification and Validation Results for Water Samples

The quality of the water sample data is excellent (Table 5-1). The percent usable data, percent unusable data, and percent completeness for the entire water dataset are 100%, 0.0%, and 97.6%, respectively.

A comparison of the validation results to the results of the electronic verification was performed during the manual validation in order to provide an indication of the accuracy of the EDV process. This comparison identified the following issues for the EDV process used for the 2017 Phase 2 RAMP water dataset:

- The EDV process did not include an evaluation of equipment blank results associated with aqueous samples. Equipment blank results are summarized in Section 5.3.
- The EDV process did not include an evaluation of the "calibration compliant" field as planned by the Phase 2 RAM QAPP (Section 12.2.1). A separate query was performed on the database to identify any instances when the calibration associated with a result was reported to be non-compliant. The query did not identify any instances of non-compliant calibrations for the data included in this DSR.

5.1.1.1 Data Verification and Validation Results for Total PCBs

The data quality for water samples analyzed for PCBs by Method 1668C (using the methods described in Section 2.1.3) is excellent (Table 5-1). The percent usable data, percent unusable data, and percent completeness for the entire PCB dataset are 100%, 0%, and 97.8%, respectively. None of the PCB data were qualified as unusable.

The EDV used to verify the PCB analytical data tracks the reason(s) that sample results are qualified for the individual assessment measures. The RAMP database was queried to determine the reason those data were qualified. However, because results from manual validation are not tracked in the RAMP database, the data validation reports were also evaluated manually. This combined assessment indicated that the EDV process identified the primary QC measures that resulted in qualification of data, as follows:

- Total PCB results summed from estimated individual PCB congener results. The Total PCB results in 100% of the samples were qualified as estimated because at least one of the individual PCB congener results that were summed to calculate the Total PCB result was qualified as estimated (including due to the standard EPA analytical data reporting convention of qualifying data as estimated when they fall between the RL and the MDL).
- Field duplicate precision. Water sample results associated with original and field duplicate samples that did not meet the project field duplicate precision criteria resulted in qualification

of positive and ND results as estimated (J and UJ, respectively) for 0.8% of the PCB dataset. A more detailed discussion of field duplicate results is presented in Section 5.2.

- Blank contamination. Positive sample results that exhibited PCB concentrations similar to that in the method blanks were qualified as ND and flagged "UB." Qualification due to blank contamination occurred for 0.3% of the PCB dataset.

As this list indicates, qualification of data for QC reasons (other than Total PCB results summed from estimated individual PCB congener results) occurred most often due to field duplicate imprecision. In addition, 19% of the data were qualified as estimated (J) due to the standard EPA analytical data reporting convention of qualifying data as estimated when they fall between the RL and the MDL.

5.1.1.2 Data Verification and Validation Results for Total Suspended Solids

The data quality for TSS by SM 2540D is good (Table 5-1). The percent usable data, percent unusable data, and percent completeness for the TSS dataset are 100%, 0%, and 78.1%, respectively. None of the TSS data were qualified as unusable.

The queries of the RAMP database and manual evaluation of the data validation reports revealed that TSS sample results were qualified for the following reasons:

- Laboratory replicate precision. Water sample results associated with original and laboratory replicate samples that did not meet the project's laboratory replicate precision criteria resulted in qualification of positive and ND results as estimated (J and UJ, respectively) for 14% of the TSS sample results.
- LCS recoveries outside of acceptance criteria. Water sample results associated with LCS recoveries outside of acceptance criteria (80% to 120%) resulted in qualification of positive and ND results as estimated (J and UJ, respectively) for 5.1% of the TSS data (nine samples).
- Exceeded holding times. A portion (2.8%) of the positive and ND TSS results were qualified as estimated (J and UJ, respectively) due to the performance of TSS analysis beyond the 7-day holding time from collection to analysis.
- Field duplicate precision. Qualification of positive results as estimated (J) due to field duplicate imprecision occurred for 0.6% of the TSS sample results (one positive sample result). A more detailed discussion of field duplicate results is presented in Section 5.2.

As shown by this list, qualification of TSS data occurred most often due to laboratory replicate imprecision.

5.1.2 Data Verification and Validation Results for Fish Tissue Samples

The overall data quality for the fish tissue sample data is excellent (Table 5-2). The percent usable data, percent unusable data, and percent completeness for the entire fish tissue dataset are 100%, 0%, and 99.4%, respectively. None of the fish data were qualified as unusable.

A comparison of the validation results to the results of the electronic verification was performed during the manual validation in order to provide an indication of the accuracy of the EDV process. One issue was identified during this comparison, which relates to the Total PCB results calculated from Aroclor or individual congener PCBs: the EDV process did not qualify the reported positive results for Total PCBs summed from estimated Aroclor results as estimated (J) when Aroclor or congener PCB results were qualified as estimated solely due to quantitation below the RLs. The impact of this issue is expected to be minimal because Total PCB results were qualified as estimated (J) if the Total PCB result was less than its RL.

5.1.2.1 Data Verification and Validation Results for Aroclor PCBs

The data quality for Aroclor PCBs in fish tissue analyzed by SW-846 Method 8082A is excellent (Table 5-2). The percent usable data, percent unusable data, and percent completeness for the entire Aroclor PCB dataset are 100%, 0%, and 99.4%, respectively. None of the data were qualified as unusable.

As noted previously, the EDV used to verify the PCB analytical data tracks the reason(s) that sample results are qualified for the individual assessment measures. The RAMP database was queried to determine the reason those data were qualified. However, because results from manual validation are not tracked in the RAMP database, the data validation reports were also evaluated manually. This combined assessment indicated that the EDV process identified the primary QC measures that resulted in qualification of data, as follows:

- Total PCB results summed from estimated individual Aroclor results. The Total PCB results in 4.0% of the samples (0.48% of the data) were qualified as estimated because at least one of the individual Aroclor results that were summed to calculate the Total PCB result was qualified as estimated.
- MS recoveries outside of acceptance criteria. Fish tissue sample results associated with surrogate recoveries outside of acceptance criteria (70% to 130%) resulted in qualification of positive results as estimated (J) for 0.1% of the Aroclor PCB data (positive results in one sample).

As this list indicates, qualification of data for QC reasons occurred most often due to Total PCB results summed from estimated individual Aroclor results. In addition, 12% of the data were qualified

as estimated (J) due to the standard EPA analytical data reporting convention of qualifying data as estimated when they fall between the RL and the MDL.

5.1.2.2 Data Verification and Validation Results for Lipid Content

The data quality for the fish tissue sample lipids content analyzed by Pace SOP S-GB-O-068-REV.01 (Attachment 2 of CAM No. 15) is excellent (Table 5-2). The percent usable data, percent unusable data, and percent completeness for the entire lipid content dataset are 100%, 0%, and 99.8%, respectively. None of the data were qualified as unusable.

As noted previously, the EDV used to verify the lipids analytical data tracks the reason(s) that sample results are qualified for the individual assessment measures. The RAMP database was queried to determine the reason those data were qualified. These queries indicated that the EDV process identified the primary QC measures that resulted in qualification of data, as follows:

- LD precision. Fish tissue sample results associated with original and LD samples that did not meet the project LD precision criteria resulted in qualification of positive and ND results as estimated (J and UJ, respectively) for 0.15% of the lipids dataset (one sample). A more detailed discussion of LD results is presented in Section 5.2.

5.2 Field and Laboratory Duplicates

Water field duplicates were generally prepared in the field for the Phase 2 RAMP at the rate of 5% of the total number of environmental samples or one per sample batch of up to 20 samples (refer to Section 3.1.3 for the specific frequency for each method). Fish tissue field duplicates were not submitted for analysis because it is impossible to collect field duplicates for fish samples. As a result, a summary of the results for the fish LDs are provided in this section (refer to Section 3.1.5 for the specific frequency for each method).

5.2.1 Aqueous Field Duplicate Results

The precision criteria for field duplicate pairs are presented in Section 10.3.1 of the Phase 2 RAM QAPP. For water field duplicate pairs where both results were greater than or equal to five times the RL, the precision criterion is that the relative percent difference (RPD) between the results should be less than or equal to 35% for PCBs by EPA Method 1668C and less than or equal to 20% for TSS. For water field duplicate pairs where at least one of the results was less than five times the RL (including when one result was ND), the precision criterion is that the difference between the results should be less than or equal to the RL. A value of one-half the RL was used for ND results in the difference calculation. If the analyte was not detected in the sample or the field duplicate sample, the RPD was not calculated and a quantitative evaluation was not made because neither sample had a positive result.

5.2.1.1 Aqueous Field Duplicate Results for PCBs

Table 5-3 presents a summary of the field duplicate results for water samples analyzed for PCBs by EPA 1668C. The table includes the following information:

- The total number of field duplicate pairs is presented in the column with the heading "Total Number Field Duplicate Pairs." The table presents the total number of field duplicate pairs for each analyte as well as the total number of field duplicate result pairs.
- The total number of field duplicate pairs that had ND results in both the parent sample and field duplicate is presented in the column with the heading "Total Number Field Duplicate Pairs with NDs for Both Samples." (All of these met field duplicate precision criteria because both results are ND.) This information is also presented by analyte.
- The total number of field duplicate pairs that had positive results in the field duplicate or parent sample is presented in the column under the heading "Total Number Field Duplicate Pairs with Positives in Either Sample." The total number, the number that met criteria, and the number that did not meet criteria, as well as the percentages that did and did not meet criteria, are presented. This information is also presented by analyte.
- The overall percentage of results that met criteria is presented in the column with the heading "Overall Percent Meet Criteria." This information is also presented by analyte.

Ten water field duplicate pairs were analyzed for PCBs by Method 1668C. A high percentage (86%) of the Total PCBs and individual congener PCB results met the field duplicate precision criteria. For Total PCBs, 80% of the results met the field duplicate precision criteria. For the individual PCB congeners, the percentage of results that met the field duplicate precision criteria ranged from 50% to 100%. The percentage of field duplicate pairs with positive results in either sample that met the field duplicate precision criteria was 81% for the individual congeners and 80% for Total PCBs. The percentage of PCB results that met field duplicate criteria were lowered by the inclusion of a field duplicate pair that was entered into the database when the original and re-extraction results of a sample did not compare well (refer to the Technical Memorandum included in Appendix A: Results of Re-Extraction of Sample OWS-THIS-T170726143612).

5.2.1.2 Aqueous Field Duplicate Results for Total Suspended Solids

Table 5-4 presents a summary of the field duplicate results for water samples analyzed for TSS. Nine field duplicate pairs were analyzed for TSS, and 89% of the results (88% of positive results) met field duplicate precision criteria.

5.2.2 Fish Laboratory Duplicate Results

The precision criteria for fish LD pairs were presented in Table A3-1 of the September 2017 revision of Attachment A to the Phase 2 RAM QAPP. For fish LD pairs where both results were greater than or

equal to five times the RL, the precision criterion was that the RPD between the results should be less than or equal to 40% for PCBs and lipids. For fish LD pairs where at least one result was less than five times the RL (including when one result was ND), the precision criterion was that the difference between the results should be less than or equal to twice the RL. A value of one-half the RL was used for ND results in the difference calculation. If the analyte was not detected in the sample or the LD, the RPD was not calculated and a quantitative evaluation was not made because neither sample had a positive result.

5.2.2.1 Fish Laboratory Duplicate Results for PCBs

A summary of the fish laboratory replicate results analyzed for Aroclor PCBs is presented in Table 5-5. The table includes the following information:

- The total number of LD pairs is presented in the column with the heading "Total Number Laboratory Duplicate Pairs." The table presents the total number of LD pairs for each analyte as well as the total number of LD result pairs.
- The total number of LD pairs that had ND results in both the parent sample and LD is presented in the column with the heading "Laboratory Duplicate Pairs with NDs for Both Samples." (All of these met LD precision criteria because both results are ND.) This information is also presented by analyte.
- The total number of LD pairs that had positive results in the LD or parent sample is presented in the column under the heading "Total Number Laboratory Duplicate Pairs with Positives in Either Sample." The total number, the number that met criteria, and the number that did not meet criteria, as well as the percentages that did and did not meet criteria, are presented. This information is also presented by analyte.
- The overall percentage of results that met criteria is presented in the column with the heading "Overall Percent Meet Criteria." This information is also presented by analyte.

A total of 56 LD pairs were analyzed for Aroclor PCBs. All results met the LD precision criteria for PCBs.

5.2.2.2 Fish Laboratory Duplicate Results for Lipids Content

A summary of the fish LD results analyzed for lipids content is presented in Table 5-6. The table includes, for each parameter and method, the same information described in Section 5.2.2.1 for Table 5-5.

A total of 56 LD pairs were analyzed for lipids content, and 98% of the positive results met LD precision criteria.

5.3 Equipment Blanks

5.3.1 Water

Equipment blanks were collected at the frequencies described in Section 3.1.2 to monitor the potential for external contamination during sample collection. Summary statistics for the results from the 2017 aqueous equipment blanks with analyte positive results greater than the MDL are presented in Table 5-7.

Trace concentrations of the individual PCB congeners and Total PCBs were detected in the eight equipment blanks collected for PCB by method 1668C analysis (Table 5-7). The maximum Total PCB concentration was 0.344 ng/L.

6 References

Anchor QEA (Anchor QEA, LLC) and ESI (Environmental Standards, Inc.), 2012. *Phase 2 Remedial Action Monitoring Quality Assurance Project Plan.* Hudson River PCBs Superfund Site. Prepared for General Electric Company, Albany, New York. May 2012.

ESI, 2017. Analytical Program Approach and Procedures. Revised Attachment A to *Phase 2 Remedial Action Monitoring Quality Assurance Project Plan*. Revised in March and again in September 2017.

QEA (Quantitative Environmental Analysis, LLC) and ESI, 2004. *Baseline Monitoring Program Quality Assurance Project Plan*. Prepared for General Electric Company, Albany, New York.

Tables

Table 2-1
2017 Water Monitoring Roles

Function	Station ID	Description	Approximate River Mile	Start Date	End Date	Sampling Frequency
Background Monitoring	WFF-BAFA	Bakers Falls Manual Station	197.1	May 24, 2017	November 21, 2017	Monthly
	WFF-ROIS	Rogers Island Manual Station	194.5	May 24, 2017	November 21, 2017	Monthly
Project Area Monitoring	WFF-THIS	Thompson Island Manual Station	187.6	May 1, 2017	December 8, 2017	Weekly
	WFF-SVDB	Schuylerville Dix Bridge Manual Sampling Location	182.7	February 26, 2017	July 3, 2017	High Flow*
	WFF-SCHU	Schuylerville Manual Sampling Location	181.3	May 1, 2017	December 8, 2017	Weekly
	WFF-WAFO	Waterford Manual Sampling Location	156.6	February 23, 2017	December 28, 2017	Weekly
Lower River Monitoring	WFF-LHAL	Lower Hudson at Albany	145	May 26, 2017	November 20, 2017	Monthly
	WFF-LHPO	Lower Hudson at Poughkeepsie	76.0	May 26, 2017	November 20, 2017	Monthly

Notes:

*The Schuylerville Dix Bridge manual sampling location was added as an additional high flow sampling location at EPA request.

Table 2-2
Water Sample Collection, Handling, and Analysis Summary

Station	Analyte	Container Specifications	Preservation	Analytical Method	Turnaround Time[1]	Holding Time[2]
Bakers Falls	PCBs	1-L amber glass bottle	Cool, 4°C +/- 2°C	EPA method 1668C	Standard	365 days to extraction, 365 days to analysis
	TSS	1-L HDPE plastic bottle	Cool, 4°C +/- 2°C	SM 2540D[3]	Standard	7 days
Rogers Island	PCBs	1-L amber glass bottle	Cool, 4°C +/- 2°C	EPA method 1668C	Standard	365 days to extraction, 365 days to analysis
	TSS	1-L HDPE plastic bottle	Cool, 4°C +/- 2°C	SM 2540D[3]	Standard	7 days
Thompson Island	PCBs	1-L amber glass bottle	Cool, 4°C +/- 2°C	EPA method 1668C	Standard	365 days to extraction, 365 days to analysis
	TSS	1-L HDPE plastic bottle	Cool, 4°C +/- 2°C	SM 2540D[3]	Standard	7 days
Schuylerville	PCBs	1-L amber glass bottle	Cool, 4°C +/- 2°C	EPA method 1668C	Standard	365 days to extraction, 365 days to analysis
	TSS	1-L HDPE plastic bottle	Cool, 4°C +/- 2°C	SM 2540D[3]	Standard	7 days
Waterford Manual	PCBs	1-L amber glass bottle	Cool, 4°C +/- 2°C	EPA method 1668C	Standard	365 days to extraction, 365 days to analysis
	TSS	1-L HDPE plastic bottle	Cool, 4°C +/- 2°C	SM 2540D[3]	Standard	7 days
Albany, Poughkeepsie	PCBs	1-L amber glass bottle	Cool, 4°C +/- 2°C	EPA method 1668C	Standard	365 days to extraction, 365 days to analysis
	TSS	1-L HDPE plastic bottle	Cool, 4°C +/- 2°C	SM 2540D[3]	Standard	7 days

Notes:

1. All turnaround times run from verified time of sample receipt at laboratory; standard TAT is 20 business days.
2. Holding times start on the date of collection.
3. Modified to be consistent with American Society for Testing and Materials Method 3977-97.

°C: degrees Celsius
EPA: U.S. Environmental Protection Agency
HDPE: high-density polyethylene
L: liter
MDL: method detection limit
PCB: polychlorinated biphenyl
TSS: total suspended solids

Table 2-3
Fish Sampling Locations and Number of Each Species per Location (2017 Spring Sampling)

Location	Size (TL) / Site Code	SMB/LMB >305 mm	BB/YB CHC/WHC >200 mm	YP/WP >170mm/>160 mm	Striped Bass >450 mm	Total	Sample Date	Schocking Seconds	Site Description	Notes
		Actual Number of Adult Fish								
Feeder Dam	FD1	20	20	20		**60**	6/13/2017	7880	Feeder Dam Pool near boat launch	
Feeder Dam Total		***20***	***20***	***20***		***60***				
Thompson Island Pool	TD1	5	2	5		**12**	6/12/2017 6/15/2017 6/19/2017	3951 3083 1244	Near Rogers Island	Extra fish collected to make up for cooler of samples lost during shipping
Thompson Island Pool	TD2	5	13	5		**23**	6/12/2017 6/19/2017	2830 1506	Near RM 193	Extra bullhead collected to make up for lack of fish at TD3; extra perch collected to make up for samples lost during shipping
Thompson Island Pool	TD3	5	0	5		**10**	6/12/2017	2137	Around three sisters islands on eastern shore	Perch not found due to lack of habitat
Thompson Island Pool	TD4	5	0	5		**10**	6/12/2017 6/15/2017 6/19/2017	2082 1227 157	Northern end of Griffin Island	Extra bass collected to make up for samples lost during shipping; bullhead not found due to lack of habitat
Thompson Island Pool*	TD5	10	15	10		**35**	6/12/2017 6/19/2017	3430 992	West channel of Griffin Island	Extra bullhead collected to make up lack of fish at TD4
TIP Totals		***30***	***30***	***30***		***90***				
Ft.Miller/Northumberland Pools (LL section)	ND1	5	10	7		**22**	6/13/2017	1922	From Thompson Island to small island below	Extra bullhead and perch collected to make up for lack of fish at ND2
Ft.Miller/Northumberland Pools (LL section)	ND2	5	0	3		**8**	6/13/2017	2238	Downstream end of pool	Bullhead not found due to lack of habitat
Ft.Miller/Northumberland Pools	ND3	5	5	5		**15**	6/15/2017	2738	Below Fort Miller dam to two small islands	
Ft.Miller/Northumberland Pools	ND5	10	10	10		**30**	6/15/2017	4776	Wetland area and island above Northumberland Dam	
FM/ND Totals		***25***	***25***	***25***		***75***				
Stillwater Pool	SW1	5	5	5		**15**	6/13/2017 6/15/2017	6987 151	Near Lock 5 and mouth of Battenkill	
Stillwater Pool	SW2	5	5	5		**15**	6/14/2017	2307	Approx. 3/4 mile usptream of Coveville	
Stillwater Pool**	SW3	10	10	10		**30**	6/14/2017	3491	Coveville	
Stillwater Pool	SW4	5	5	5		**15**	6/14/2017	2732	Near RM 173	
Stillwater Pool	SW5	5	5	5		**15**	6/14/2017	2671	Just above Stillwater Dam	
SW Totals		***30***	***30***	***30***		***90***				
Albany/Troy	AT1	20	20	20	20	**80**	5/19/2017 6/16/2017 6/20/2017	4152 14786 1121	Below Federal Dam	Striped bass collected on 5/19; perch collection includes 10 YP and 10 WP
Albany/Troy Totals		***20***	***20***	***20***	***20***	***80***				
Catskill	CS		20	20	20	**60**	5/18/2017 6/20/2017	6431 7700	Between mouth of Catskill Creek and Rip Van Winkle Bridge	Black bass not targeted; perch collection included 10 YP and 10 WP
Catskill Totals			***20***	***20***	***20***	***60***				
Tappan Zee	TZ				20	**20**	4/18/2017 4/19/2017 4/20/2017	NA	Between Tappan Zee Bridge and Piermont Point	Fourteen striped bass collected angling and 6 striped bass collected from charter captain's permitted bait net
Tappan Zee Totals					***20***	***20***				

Notes:
Fish that were recollected in Thompson Island Pool due to shipping error not shown.
*Historical DEC location behind Griffin Island
**Historical DEC location near Coveville
SMB/LMB: equal numbers from each location when possible
YP/WP: equal numbers of each at Albany/Troy (10 of each) when possible
RM: river mile
TL: total length
RAMP: Remedical Action Monitoring Program
LL: Landlocked Section

Table 2-4
Fish Sampling Locations and Number of Each Species per Location (2017 Fall Sampling)

Location	Size (TL)	PKSD 70-130 mm	STS[1]	Total	Sample Date	Shocking Seconds	Site description	Notes
	Site Code	Number of Fish[2]						
Feeder Dam	FD1	20	10	**30**	8/29/2017	1926	Feeder Dam pool near boat launch	Collected all targeted numbers
Feeder Dam Total		***20***	***10***	***30***				
Thompson Island Pool	TD1	5	2	**7**	8/28/2017	202	Near Rogers Island	Collected all targeted numbers
Thompson Island Pool	TD2	5	2	**7**	8/28/2017	2090	Near RM 193	Collected all targeted numbers
Thompson Island Pool	TD3	5	2	**7**	8/28/2017	1166	Around three sisters islands on eastern shore	Collected all targeted numbers
Thompson Island Pool	TD4	5	2	**7**	8/28/2017	1151	Northern end of Griffin Island	Collected all targeted numbers
Thompson Island Pool*	TD5	10	2	**12**	8/28/2017	2991	Near RM 190 - along eastern shoreline	Collected all targeted numbers
TIP Totals		***30***	***10***	***40***				
Fort Miller/Northumberland Pools (LL section)	ND1	6	2	**8**	8/28/2017	1537	From Thompson Island to small island below	Collected extra PKSD to make up for ND2
Fort Miller/Northumberland Pools (LL section)	ND2	4	2	**6**	8/28/2017	4806	Downstream end of pool	Four out of 5 PKSD Collected
Fort Miller/Northumberland Pools	ND3	5	2	**7**	8/29/2017	2071	Below Fort Miller dam to two small islands	Collected all targeted numbers
Fort Miller/Northumberland Pools	ND4	0	0	**0**			Abandoned	
Fort Miller/Northumberland Pools	ND5	10	4	**14**	8/29/2017	717	Wetland area above Northumberland Dam	Collected all targeted numbers
FM/ND Totals		***25***	***10***	***35***				
Stillwater Pool	SW1	5	2	**7**	8/29/2017	605	Below Battenkill	Collected all targeted numbers
Stillwater Pool	SW2	5	2	**7**	8/29/2017	305	Approximately 3/4 mile usptream of Coveville	Collected all targeted numbers
Stillwater Pool	SW3	5	2	**7**	8/29/2017	1534	Coveville	Collected all targeted numbers
Stillwater Pool	SW4	5	2	**7**	8/30/2017	255	Near RM 173	Collected all targeted numbers
Stillwater Pool**	SW5	10	2	**12**	8/30/2017	545	Just above Stillwater Dam	Collected all targeted numbers
SW Totals		***30***	***10***	***40***				
Albany/Troy	AT1	20	10	**30**	8/30/2017	7784	Between Dunn Memorial Bridge and Route 90 Bridge	Collected all targeted numbers
Albany/Troy Totals		***20***	***10***	***30***				
Total by Species		***125***	***50***	***175***				

Notes:

1. Substitute species for Spottail Shiner include: Mimic Shiner, Bluntnose Minnow, Golden Shiner, Spotfin Shiner, Banded Killifish and Fallfish
2. Number of composite samples for forage fish

*Historical DEC location across from Griffin Island (east channel)

**Historical DEC location near Stillwater Dam

DEC: Department of Environmental Conservation

mm: millimeter

PKSD: Pumpkinseed

RAMP: Remedical Action Monitoring Program

LL: Landlocked Section

RM: river mile

STS: Spottail Shiner

TL: total length

Table 3-1

Summary of Percentage of Validated 2017 RAMP Water and Fish Data

Analysis Fraction	Total ENV Samples		
	Number of ENV Samples	Number of ENV Samples Validated	Percent ENV Samples Validated
Congener PCB in water (1668C, Vista SOP 31, Rev 15)	176	14	8.0%
Total suspended solids in water (SM 2540D)	174	17	9.8%
Aroclor PCBs in fish tissue (SW-846 8082A, S-GB-O-067-Rev.00)	650	35	5.4%

Notes:
ENV: environmental
PCB: polychlorinated biphenyl
RAMP: Remedial Action Monitoring Program

Table 4-1
Total PCB and TSS Summary Statistics for Water

Location	Sample Counts		Detect	Non-detect	Detected Concentrations		
	ENV	DUP			Minimum	Average	Maximum
Total PCBs (ng/L) Off-season							
Bakers Falls	7	0	7	0	0.27	0.71	1.89
Rogers Island	7	1	8	0	0.51	0.68	0.94
Thompson Island	29	2	31	0	1.62	8.33	32.6
Schuylerville	29	1	30	0	4.11	12.0	36.4
Waterford	43	2	45	0	2.67	11.4	18.6
Albany	7	0	7	0	5.13	8.99	13.1
Poughkeepsie	7	1	8	0	7.47	9.06	12.1
TSS (mg/L) Off-season							
Bakers Falls	7	0	6	1	1.40	2.12	4.50
Rogers Island	7	1	6	2	1.20	1.58	2.40
Thompson Island	29	1	28	2	1.10	5.73	68.7
Schuylerville	29	1	30	0	1.00	3.62	18.2
Waterford	43	2	45	0	1.60	10.0	56.4
Albany	7	0	7	0	3.60	18.6	74.8
Poughkeepsie	7	1	8	0	9.30	13.8	19.8
Total PCBs (ng/L) High Flow							
Schuylerville Dix Bridge	26	1	27	0	3.03	13.1	37.7
Waterford	21	2	23	0	3.71	17.7	37.3
TSS (mg/L) High Flow							
Schuylerville Dix Bridge	26	1	27	0	4.40	25.6	106
Waterford	21	2	23	0	12.7	75.7	283

Notes:

Duplicate samples are averaged with parent samples.

Statistics are based on detected results only.

DUP: duplicate

ENV: environmental

mg/L: milligrams per liter

ng/L: nanograms per liter

PCB: polychlorinated biphenyl

TSS: total suspended solids

Table 4-2
Water Quality Parameter Summary Statistics

Location	Parameter	Minimum	Average	Maximum	Units
Bakers Falls	Dissolved Oxygen	7.94	9.64	12.8	mg/L
	pH	7.04	7.62	8.23	pH
	Specific Conductance	0.04	0.09	0.12	µs/cm
	Turbidity	0.20	1.83	4.50	NTU
	Water Temperature	7.42	18.3	25.4	°C
Rogers Island	Dissolved Oxygen	7.83	9.74	13.0	mg/L
	pH	6.71	7.49	8.19	pH
	Specific Conductance	0.06	0.09	0.12	µs/cm
	Turbidity	0.40	2.42	5.40	NTU
	Water Temperature	6.44	17.4	24.1	°C
Thompson Island	Dissolved Oxygen	7.62	9.42	14.4	mg/L
	pH	6.55	7.45	8.24	pH
	Specific Conductance	0.05	0.09	0.13	µs/cm
	Turbidity	0.20	3.39	17.1	NTU
	Water Temperature	4.45	17.6	24.5	°C
Schuylerville Dix Bridge	Dissolved Oxygen	8.69	12.4	15.5	mg/L
	pH	6.76	7.82	9.26	pH
	Specific Conductance	0.05	0.08	0.12	µs/cm
	Turbidity	2.30	18.8	97.2	NTU
	Water Temperature	2.36	7.84	21.2	°C
Schuylervile Manual	Dissolved Oxygen	7.75	9.37	13.7	mg/L
	pH	6.88	7.54	8.37	pH
	Specific Conductance	0.06	0.11	0.15	µs/cm
	Turbidity	0.20	4.87	14.5	NTU
	Water Temperature	4.17	18.6	24.5	°C
Waterford	Dissolved Oxygen	7.81	10.7	16.1	mg/L
	pH	6.55	7.42	8.41	pH
	Specific Conductance	0.07	0.15	0.30	µs/cm
	Turbidity	0.20	13.8	123	NTU
	Water Temperature	0.46	14.3	25.9	°C
Albany	Dissolved Oxygen	7.25	9.60	12.8	mg/L
	pH	6.81	7.69	8.16	pH
	Specific Conductance	0.15	0.22	0.27	µs/cm
	Turbidity	2.70	15.6	39.0	NTU
	Water Temperature	5.68	17.3	24.4	°C

Table 4-2
Water Quality Parameter Summary Statistics

Location	Parameter	Minimum	Average	Maximum	Units
Poughkeepsie	Dissolved Oxygen	5.95	8.63	11.0	mg/L
	pH	7.11	7.63	8.06	pH
	Specific Conductance	0.18	0.24	0.27	µs/cm
	Turbidity	1.20	9.63	18.3	NTU
	Water Temperature	7.86	19.1	25.4	°C

Notes:
°C: degrees Celsius
µs/cm: microsiemens per centimeter
mg/L: milligrams per liter
NTU: nephelometric turbidity units

Table 4-3
Aroclor PCB Summary Statistics for Black Bass

Species	Pool	Station Number	Count	Average (mg/kg)	Minimum (mg/kg)	Maximum (mg/kg)	2 SE (mg/kg)
Largemouth Bass	Feeder Dam	1	6	0.06	ND	0.11	0.03
	Thompson Island Pool	2	1	0.22	0.22	0.22	-
	Thompson Island Pool	5	8	1.51	0.09	4.30	0.91
	Northumberland/Fort Miller	5	4	1.61	0.37	4.75	2.10
	Stillwater	1	2	0.93	0.55	1.32	0.77
	Stillwater	2	4	1.20	0.28	2.50	0.94
	Stillwater	3	10	1.59	0.27	2.98	0.57
	Stillwater	4	3	0.43	0.19	0.86	0.43
	Stillwater	5	5	1.53	0.61	2.49	0.78
Smallmouth Bass	Feeder Dam	1	8	0.08	0.01	0.26	0.06
	Thompson Island Pool	1	5	2.39	1.44	3.52	0.93
	Thompson Island Pool	2	4	0.62	0.25	0.91	0.31
	Thompson Island Pool	3	5	2.63	1.02	4.28	1.24
	Thompson Island Pool	4	5	0.93	0.31	1.97	0.57
	Thompson Island Pool	5	2	1.01	0.74	1.28	0.54
	Northumberland/Fort Miller	1	5	1.82	1.59	2.04	0.18
	Northumberland/Fort Miller	2	5	2.23	0.87	5.65	1.74
	Northumberland/Fort Miller	3	5	0.81	0.47	1.36	0.33
	Northumberland/Fort Miller	5	6	1.86	1.16	3.08	0.58
	Stillwater	1	3	0.71	0.05	1.10	0.67
	Stillwater	2	1	2.42	2.42	2.42	-
	Stillwater	4	2	1.84	1.69	1.98	0.29
	Albany/Troy	1	20	2.13	0.45	4.03	0.56

Notes:
Prep – fillet
Non-detect values were set to half of the method detection limit to calculate average and 2 SE.
mg/kg: milligrams per kilogram
ND: non-detect
PCB: polychlorinated biphenyl
SE: Standard Error

Table 4-4

Aroclor PCB Summary Statistics for Ictalurids

Species	Pool	Station Number	Count	Average (mg/kg)	Minimum (mg/kg)	Maximum (mg/kg)	2 SE (mg/kg)
Brown Bullhead	Feeder Dam	1	15	0.07	ND	0.21	0.03
	Thompson Island Pool	1	3	1.11	0.27	2.33	1.25
		2	9	0.59	0.15	1.65	0.31
		5	13	1.11	0.32	2.60	0.36
	Northumberland/Fort Miller	1	9	3.22	0.56	9.84	1.91
		3	5	2.51	0.58	4.51	1.37
		5	9	1.15	0.16	2.45	0.49
	Stillwater	1	5	0.67	0.31	1.04	0.24
		2	5	0.88	0.51	1.21	0.24
		3	10	0.96	0.26	1.69	0.34
		4	4	0.66	0.23	1.14	0.37
		5	4	1.57	1.06	2.05	0.43
	Catskill	1	20	0.33	0.08	0.66	0.06
Channel Catfish	Albany/Troy	1	20	1.62	0.46	2.45	0.25
Yellow Bullhead	Feeder Dam	1	1	0.02	0.02	0.02	-
	Thompson Island Pool	1	2	1.11	0.98	1.23	0.25
	Thompson Island Pool	2	1	0.14	0.14	0.14	-
	Thompson Island Pool	5	2	0.70	0.41	0.99	0.58
	Northumberland/Fort Miller	1	1	0.42	0.42	0.42	-
	Northumberland/Fort Miller	5	1	0.37	0.37	0.37	-
	Stillwater	4	1	0.16	0.16	0.16	-
	Stillwater	5	1	0.20	0.20	0.20	-

Notes:

Prep – fillet

Non-detect values were set to half of the method detection limit to calculate average and 2 SE.

mg/kg: milligrams per kilogram

ND: non-detect

PCB: polychlorinated biphenyl

SE: Standard Error

Table 4-5
Aroclor PCB Summary Statistics for Perch

Species	Pool	Station Number	Count	Average (mg/kg)	Minimum (mg/kg)	Maximum (mg/kg)	2 SE (mg/kg)
White perch	Albany/Troy	1	10	1.02	0.32	2.15	0.40
	Catskill	1	10	0.36	0.14	0.55	0.09
Yellow perch	Feeder Dam	1	11	0.04	ND	0.07	0.01
	Thompson Island Pool	1	5	0.97	0.24	1.73	0.51
		2	5	0.38	0.18	0.77	0.21
		3	5	1.79	1.17	2.36	0.40
		4	5	0.47	0.17	1.10	0.33
		5	10	0.25	0.09	0.36	0.05
	Northumberland/Fort Miller	1	7	0.93	0.45	1.94	0.41
		2	3	1.04	0.62	1.55	0.55
		3	5	1.05	0.33	1.90	0.56
		5	10	1.04	0.57	1.75	0.26
	Stillwater	1	5	0.41	0.29	0.60	0.12
		2	5	0.56	0.41	0.88	0.17
		3	10	0.77	0.24	1.55	0.26
		4	5	0.52	0.31	1.04	0.27
		5	5	0.92	0.44	1.36	0.36
	Albany/Troy	1	10	0.37	0.21	0.60	0.09
	Catskill	1	10	0.23	0.08	0.38	0.06

Notes:
Prep – fillet
Non-detect values were set to half of the method detection limit to calculate average and 2 SE.
mg/kg: milligrams per kilogram
ND: non-detect
PCB: polychlorinated biphenyl
SE: Standard Error

Table 4-6

Aroclor PCB Summary Statistics for Striped Bass

Pool	Station Number	Count	Average (mg/kg)	Minimum (mg/kg)	Maximum (mg/kg)	2 SE (mg/kg)
Albany/Troy	1	20	0.90	0.15	6.07	0.64
Catskill	1	20	0.67	0.11	4.58	0.47
Tappan Zee	1	20	0.31	0.08	0.76	0.08

Notes:

Prep – fillet

Non-detect values were set to half of the method detection limit to calculate average and 2 SE.

mg/kg: milligrams per kilogram

PCB: polychlorinated biphenyl

SE: Standard Error

Table 4-7

Aroclor PCB Summary Statistics for Pumpkinseed

Pool	Station Number	Count	Average (mg/kg)	Minimum (mg/kg)	Maximum (mg/kg)	2 SE (mg/kg)
Feeder Dam	1	20	0.06	0.03	0.09	0.01
Thompson Island Pool	1	5	7.46	4.08	12.80	2.88
	2	5	1.48	0.87	2.34	0.64
	3	5	36.60	10.00	93.20	29.37
	4	5	1.19	0.52	1.87	0.45
	5	10	1.95	1.37	2.65	0.24
Northumberland/Fort Miller	1	6	5.36	1.92	12.50	3.25
	2	4	10.76	6.09	15.20	3.98
	3	5	3.89	1.85	8.17	2.23
	5	10	3.81	2.38	6.44	0.73
Stillwater	1	5	5.99	4.67	8.04	1.48
	2	5	2.97	1.87	4.77	0.99
	3	5	1.04	0.38	1.48	0.51
	4	5	1.70	1.45	2.01	0.19
	5	10	1.84	1.41	2.50	0.23
Albany/Troy	1	20	0.66	0.27	0.88	0.07

Notes:

Prep – whole body

Non-detect values were set to half of the method detection limit to calculate average and 2 SE.

mg/kg: milligrams per kilogram

PCB: polychlorinated biphenyl

SE: Standard Error

Table 4-8

Aroclor PCB Summary Statistics for Forage Fish

Pool	Station Number	Count	Average (mg/kg)	Minimum (mg/kg)	Maximum (mg/kg)	2 SE (mg/kg)
Feeder Dam	1	10	0.08	0.05	0.12	0.01
Thompson Island Pool	1	2	1.08	1.01	1.15	0.14
	2	2	1.31	0.61	2.00	1.39
	3	2	3.78	3.30	4.25	0.95
	4	2	1.18	1.03	1.33	0.30
	5	2	1.38	1.26	1.50	0.24
Northumberland/Fort Miller	1	2	4.18	2.26	6.10	3.84
	2	2	4.45	3.42	5.47	2.05
	3	2	3.71	3.68	3.74	0.06
	5	4	3.93	3.28	4.64	0.58
Stillwater	1	2	7.07	6.11	8.03	1.92
	2	2	1.36	1.27	1.44	0.17
	3	2	1.39	1.11	1.66	0.55
	4	2	1.97	1.60	2.34	0.74
	5	2	2.89	1.68	4.09	2.41
Albany/Troy	1	10	0.72	0.37	1.32	0.18

Notes:

Prep – whole body (composite)

Non-detect values were set to half of the method detection limit to calculate average and 2 SE.

Forage Fish = Golden Shiner, Spottail Shiner, Fallfish, Spotfin Shiner, Mimic Shiner, or Banded Killifish

mg/kg : milligrams per kilogram

PCB: polychlorinated biphenyl

SE: Standard Error

Table 4-9

Percent Lipid Summary Statistics for Black Bass

Species	Pool	Station Number	Count	Average %	Minimum %	Maximum %	2 SE %
Largemouth Bass	Feeder Dam	1	12	0.44	0.19	1.50	0.20
	Thompson Island Pool	2	1	0.27	0.27	0.27	-
		5	8	0.41	0.28	0.78	0.11
	Northumberland/Fort Miller	5	4	0.40	0.21	0.64	0.20
	Stillwater	1	2	0.23	0.20	0.25	0.05
		2	4	0.45	0.25	0.63	0.16
		3	10	0.40	0.17	1.20	0.20
		4	3	0.31	0.29	0.33	0.02
		5	5	0.59	0.28	0.99	0.31
Smallmouth Bass	Feeder Dam	1	8	0.31	0.20	0.36	0.04
	Thompson Island Pool	1	5	0.52	0.27	0.83	0.18
		2	4	0.44	0.32	0.65	0.15
		3	5	0.45	0.16	0.70	0.19
		4	5	0.64	0.41	1.10	0.24
		5	2	0.68	0.62	0.74	0.12
	Northumberland/Fort Miller	1	5	0.45	0.35	0.58	0.09
		2	5	0.33	0.25	0.41	0.06
		3	5	0.34	0.18	0.48	0.11
		5	6	0.42	0.30	0.52	0.08
	Stillwater	1	3	0.36	0.23	0.56	0.20
		2	1	1.00	1.00	1.00	-
		4	2	0.40	0.32	0.47	0.15
	Albany/Troy	1	20	1.02	0.34	2.80	0.34

Notes:

Prep – fillet

SE: Standard Error

Table 4-10

Percent Lipid Summary Statistics for Ictalurids

Species	Pool	Station Number	Count	Average %	Minimum %	Maximum %	2 SE %
Brown Bullhead	Feeder Dam	1	19	0.61	0.35	1.50	0.14
	Thompson Island Pool	1	3	1.25	0.84	1.90	0.66
		2	9	0.88	0.36	2.00	0.39
		5	13	0.85	0.33	1.50	0.21
	Northumberland/Fort Miller	1	9	0.95	0.43	1.40	0.20
		3	5	1.40	0.39	2.00	0.59
		5	9	0.99	0.47	1.80	0.26
	Stillwater	1	5	0.85	0.60	1.40	0.30
		2	5	0.77	0.58	1.10	0.18
		3	10	0.69	0.32	1.30	0.22
		4	4	0.89	0.36	1.40	0.45
		5	4	1.15	0.89	1.30	0.18
	Catskill	1	20	1.35	0.67	2.70	0.25
Channel Catfish	Albany/Troy	1	20	3.36	0.76	7.10	0.73
Yellow Bullhead	Feeder Dam	1	1	1.00	1.00	1.00	-
	Thompson Island Pool	1	2	0.66	0.41	0.91	0.50
	Thompson Island Pool	2	1	0.47	0.47	0.47	-
	Thompson Island Pool	5	2	0.63	0.39	0.86	0.47
	Northumberland/Fort Miller	1	1	0.52	0.52	0.52	-
	Northumberland/Fort Miller	5	1	0.82	0.82	0.82	-
	Stillwater	4	1	0.32	0.32	0.32	-
	Stillwater	5	1	0.34	0.34	0.34	-

Notes:

Prep – fillet

SE: Standard Error

Table 4-11

Percent Lipid Summary Statistics for Perch

Species	Pool	Station Number	Count	Average %	Minimum %	Maximum %	2 SE %
White Perch	Albany/Troy	1	10	1.23	0.77	1.60	0.21
	Catskill	1	10	0.70	0.28	1.20	0.19
Yellow Perch	Feeder Dam	1	20	0.91	0.45	1.50	0.13
	Thompson Island Pool	1	5	0.85	0.33	1.10	0.27
		2	5	0.87	0.52	1.40	0.30
		3	5	0.92	0.32	1.30	0.34
		4	5	0.78	0.21	1.70	0.50
		5	10	0.49	0.25	0.78	0.12
	Northumberland/Fort Miller	1	7	0.80	0.44	1.10	0.23
		2	3	0.92	0.39	1.40	0.59
		3	5	1.02	0.27	1.80	0.53
		5	10	1.49	0.54	2.70	0.46
	Stillwater	1	5	0.85	0.61	1.30	0.25
		2	5	0.82	0.53	1.20	0.23
		3	10	1.09	0.39	1.50	0.23
		4	5	1.11	0.64	1.80	0.39
		5	5	1.29	0.75	2.10	0.52
	Albany/Troy	1	10	0.61	0.29	1.30	0.18
	Catskill	1	10	0.49	0.11	0.92	0.14

Notes:

Prep – fillet

SE: Standard Error

Table 4-12

Percent Lipid Summary Statistics for Striped Bass

Pool	Station Number	Count	Average %	Minimum %	Maximum %	2 SE %
Albany/Troy	1	20	2.36	0.49	10.00	1.06
Catskill	1	20	1.75	0.38	4.20	0.53
Tappan Zee	1	20	2.51	0.34	6.20	0.66

Notes:

Prep – fillet

SE: Standard Error

Table 4-13
Percent Lipid Summary Statistics for Pumpkinseed

Pool	Station Number	Count	Average %	Minimum %	Maximum %	2 SE %
Feeder Dam	1	20	2.74	2.10	3.50	0.16
Thompson Island Pool	1	5	3.44	3.00	3.90	0.33
	2	5	2.92	2.20	3.60	0.47
	3	5	3.06	2.20	3.60	0.49
	4	5	2.86	2.60	3.20	0.22
	5	10	2.70	2.10	3.60	0.31
Northumberland/Fort Miller	1	6	3.18	2.00	4.60	0.74
	2	4	3.60	3.10	4.00	0.37
	3	5	2.70	2.10	3.00	0.33
	5	10	2.97	2.20	3.60	0.30
Stillwater	1	5	2.62	1.80	3.10	0.44
	2	5	2.54	2.00	4.00	0.75
	3	5	2.32	2.00	2.50	0.17
	4	5	2.82	2.50	3.00	0.18
	5	10	2.68	2.30	3.50	0.23
Albany/Troy	1	20	2.12	0.83	3.50	0.23

Notes:
Prep – whole body
SE: Standard Error

Table 4-14

Percent Lipid Summary Statistics for Forage Fish

Pool	Station Number	Count	Average %	Minimum %	Maximum %	2 SE %
Feeder Dam	1	10	3.60	1.90	4.70	0.65
Thompson Island Pool	1	2	4.30	4.00	4.60	0.60
	2	2	3.90	3.10	4.70	1.60
	3	2	5.05	5.00	5.10	0.10
	4	2	5.30	5.00	5.60	0.60
	5	2	1.90	1.70	2.10	0.40
Northumberland/Fort Miller	1	2	3.50	2.50	4.50	2.00
	2	2	3.20	2.70	3.70	1.00
	3	2	4.45	4.30	4.60	0.30
	5	4	4.73	4.50	5.00	0.22
Stillwater	1	2	3.35	1.70	5.00	3.30
	2	2	2.50	2.40	2.60	0.20
	3	2	2.75	1.80	3.70	1.90
	4	2	3.30	3.10	3.50	0.40
	5	2	5.40	4.40	6.40	2.00
Albany/Troy	1	10	3.62	2.00	5.60	0.82

Notes:

Prep – whole body (composite)

Forage fish = Golden Shiner, Spottail Shiner, Fallfish, Spotfin Shiner, Mimic Shiner, or Banded Killifish

SE: Standard Error

Table 4-15
Fish Sex Summary

Species	Pool	Total Count	Count of Males	Count of Females	Count of Unknowns
Largemouth Bass	Feeder Dam	12	4	8	0
	Thompson Island Pool	9	7	2	0
	Northumberland/Fort Miller	4	1	3	0
	Stillwater	24	18	6	0
Smallmouth Bass	Feeder Dam	8	7	1	0
	Thompson Island Pool	21	9	12	0
	Northumberland/Fort Miller	21	14	7	0
	Stillwater	6	4	2	0
	Albany/Troy	20	9	11	0
Brown Bullhead	Feeder Dam	19	5	14	0
	Thompson Island Pool	25	6	19	0
	Northumberland/Fort Miller	23	4	12	7
	Stillwater	28	7	21	0
	Catskill	20	5	13	2
Channel Catfish	Albany/Troy	20	18	2	0
Yellow Bullhead	Feeder Dam	1	1	0	0
	Thompson Island Pool	5	1	3	1
	Northumberland/Fort Miller	2	0	2	0
	Stillwater	2	0	1	1
White Perch	Albany/Troy	10	5	4	1
	Catskill	10	4	3	3
Yellow Perch	Feeder Dam	20	20	0	0
	Thompson Island Pool	30	29	1	0
	Northumberland/Fort Miller	25	14	2	9
	Stillwater	30	26	2	2
	Albany/Troy	10	5	0	5
	Catskill	10	3	0	7
Striped Bass	Albany/Troy	20	8	12	0
	Catskill	20	12	8	0
	Tappan Zee	20	10	9	1

Table 5-1

Summary of Analytical Data Quality for 2017 Aqueous Environmental Samples[1]

Analysis Fraction	Number of Results Qualified[2]										Total Number of Results[4]	Percent Completeness[5]	Percent Unusable Data[6]	Percent Usable Data[7]	Qualitative Data Quality
	Unqualified Positive Results	U	UM	UB	JN	J	J[3]	UJ	R	UR					
Congener PCBs (EPA 1668C)	14,591	8,545	1,767	93	0	6,506	6,034	2	0	0	31,504	97.8%	0.0%	100.0%	Excellent
Total suspended solids (SM 2540D)	117	2	NA	0	0	55	NA	2	0	0	176	67.6%	0.0%	100.0%	Above Average
Entire water sample dataset	14,708	8,547	1,767	93	0	6,561	6,034	4	0	0	31,680	97.6%	0.0%	100.0%	Excellent

Notes:

1. Summary is for water environmental samples and does not include results from field duplicates, field blanks, lab duplicates, matrix spikes, or blanks. Summary is based on qualification of data from verification and validation.

2. Results are the number of individual analytes in the analysis fraction. For example, there are 179 analytes in the PCB congener analysis by EPA 1668C.

3. Results qualified as estimates due to being below the reporting limit. For example, of the 6,504 PCB congener results that were qualified J, 6,034 results were qualified J due to being below the reporting limit.

4. Total number of results is the summation of all qualified and unqualified results.

5. The % completeness is the sum of results that were valid as reported [unqualified positive results + U]/total number of results - J[3].

6. The % unusable data is the sum of the results qualified R + UR/total number of results.

7. The % usable data is the sum of the unqualified positive results + U + UM + UB + J + JN + UJ/total number of results.

PCB: polychlorinated biphenyl

Table 5-2

Summary of Analytical Data Quality for 2017 Fish Tissue Environmental Samples[1]

Analysis Fraction	Number of Results Qualified[2]									Total Number of Results[4]	Percent Completeness[5]	Percent Unusable Data[6]	Percent Usable Data[7]	Qualitative Data Quality
	Unqualified Positive Results	U	U*	JN	J	J[3]	UJ	R	UR					
Aroclor PCBs (SW-846 8082A)	1,964	2,581	0	0	659	630	0	0	0	5,204	99.4%	0.0%	100%	Excellent
Lipids (S-GB-O-068-REV.01)	649	0	0	0	1	NA	0	0	0	650	99.8%	0.0%	100%	Excellent
Entire fish tissue dataset	2,613	2,581	0	0	660	630	0	0	0	5,854	99.4%	0.0%	100%	Excellent

Notes:

1. Summary is for fish tissue environmental samples and does not include results from lab duplicates, matrix spikes or blanks. Summary is based on qualification of data from verification and validation.

2. Results are the number of individual analytes in the analysis fraction. For example, there are eight analytes in the Total PCBs as Aroclors analysis fraction.

3. Results qualified as estimates due to being below the reporting limit. For example, of the ## SW-846 8082A results that were qualified J,## results were qualified J due to being below the reporting limit.

4. Total number of results is the summation of all qualified and unqualified results.

5. The % completeness is the sum of results that were valid as reported [Unqualified Positive Results + U]/Total Number of Results - J[4].

6. The % unusable data is the sum of the results qualified R + UR/total number of results.

7. The % usable data is the sum of the unqualified positive results + U + U* + J + JN + UJ/total number of results.

PCB: polychlorinated biphenyl

RAMP: Remedial Action Monitoring Program

Table 5-3

Summary of Water Field Duplicate Results for Total PCBs by EPA Method 1668C in 2017

Method	Matrix	Analyte	Total Number Field Duplicate Pairs	Total Number Field Duplicate Pairs with NDs for Both Samples	Total Number Field Duplicate Pairs with Positives in Either Sample					Overall Percent Meet Criteria
					Total Number	Number Meet Criteria	Number Do Not Meet Criteria	Percent Meet Criteria	Percent Do Not Meet Criteria	
EPA 1668C	Water	Total PCB	10	0	10	8	2	80	20	80
EPA 1668C	Water	Decachlorobiphenyl	10	1	9	5	4	56	44	60
EPA 1668C	Water	Dichlorobiphenyl	10	0	10	8	2	80	20	80
EPA 1668C	Water	Heptachlorobiphenyl	10	0	10	7	3	70	30	70
EPA 1668C	Water	Hexachlorobiphenyl	10	0	10	8	2	80	20	80
EPA 1668C	Water	Monochlorobiphenyl	10	1	9	7	2	78	22	80
EPA 1668C	Water	Nonachlorobiphenyl	10	1	9	4	5	44	56	50
EPA 1668C	Water	Octachlorobiphenyl	10	1	9	4	5	44	56	50
EPA 1668C	Water	Pentachlorobiphenyl	10	0	10	8	2	80	20	80
EPA 1668C	Water	Tetrachlorobiphenyl	10	0	10	8	2	80	20	80
EPA 1668C	Water	Trichlorobiphenyl	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-1	10	1	9	7	2	78	22	80
EPA 1668C	Water	PCB-2	10	2	8	6	2	75	25	80
EPA 1668C	Water	PCB-3	10	1	9	7	2	78	22	80
EPA 1668C	Water	PCB-4/10	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-5/8	10	1	9	7	2	78	22	80
EPA 1668C	Water	PCB-6	10	2	8	5	3	63	38	70
EPA 1668C	Water	PCB-7/9	10	4	6	4	2	67	33	80
EPA 1668C	Water	PCB-11	10	4	6	4	2	67	33	80
EPA 1668C	Water	PCB-12/13	10	2	8	6	2	75	25	80
EPA 1668C	Water	PCB-14	10	10	0	0	0	NA	NA	100
EPA 1668C	Water	PCB-15	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-16/32	10	0	10	7	3	70	30	70
EPA 1668C	Water	PCB-17	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-18	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-19	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-20/21/33	10	0	10	8	2	80	20	80

Table 5-3

Summary of Water Field Duplicate Results for Total PCBs by EPA Method 1668C in 2017

Method	Matrix	Analyte	Total Number Field Duplicate Pairs	Total Number Field Duplicate Pairs with NDs for Both Samples	Total Number Field Duplicate Pairs with Positives in Either Sample					Overall Percent Meet Criteria
					Total Number	Number Meet Criteria	Number Do Not Meet Criteria	Percent Meet Criteria	Percent Do Not Meet Criteria	
EPA 1668C	Water	PCB-22	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-23	10	5	5	5	0	100	0	100
EPA 1668C	Water	PCB-24/27	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-25	10	0	10	7	3	70	30	70
EPA 1668C	Water	PCB-26	10	0	10	7	3	70	30	70
EPA 1668C	Water	PCB-28	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-29	10	5	5	5	0	100	0	100
EPA 1668C	Water	PCB-30	10	5	5	5	0	100	0	100
EPA 1668C	Water	PCB-31	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-34	10	1	9	6	3	67	33	70
EPA 1668C	Water	PCB-35	10	1	9	7	2	78	22	80
EPA 1668C	Water	PCB-36	10	5	5	4	1	80	20	90
EPA 1668C	Water	PCB-37	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-38	10	1	9	7	2	78	22	80
EPA 1668C	Water	PCB-39	10	1	9	7	2	78	22	80
EPA 1668C	Water	PCB-40	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-41/64/71/72	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-42/59	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-43/49	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-44	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-45	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-46	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-47	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-48/75	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-50	10	2	8	7	1	88	13	90
EPA 1668C	Water	PCB-51	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-52/69	10	0	10	8	2	80	20	80

Table 5-3

Summary of Water Field Duplicate Results for Total PCBs by EPA Method 1668C in 2017

Method	Matrix	Analyte	Total Number Field Duplicate Pairs	Total Number Field Duplicate Pairs with NDs for Both Samples	Total Number Field Duplicate Pairs with Positives in Either Sample					Overall Percent Meet Criteria
					Total Number	Number Meet Criteria	Number Do Not Meet Criteria	Percent Meet Criteria	Percent Do Not Meet Criteria	
EPA 1668C	Water	PCB-53	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-54	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-55	10	2	8	6	2	75	25	80
EPA 1668C	Water	PCB-56/60	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-57	10	1	9	7	2	78	22	80
EPA 1668C	Water	PCB-58	10	2	8	8	0	100	0	100
EPA 1668C	Water	PCB-61/70	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-62	10	10	0	0	0	NA	NA	100
EPA 1668C	Water	PCB-63	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-65	10	3	7	7	0	100	0	100
EPA 1668C	Water	PCB-66/76	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-67	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-68	10	0	10	7	3	70	30	70
EPA 1668C	Water	PCB-73	10	1	9	7	2	78	22	80
EPA 1668C	Water	PCB-74	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-77	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-78	10	9	1	1	0	100	0	100
EPA 1668C	Water	PCB-79	10	2	8	7	1	88	13	90
EPA 1668C	Water	PCB-80	10	10	0	0	0	NA	NA	100
EPA 1668C	Water	PCB-81	10	10	0	0	0	NA	NA	100
EPA 1668C	Water	PCB-82	10	1	9	7	2	78	22	80
EPA 1668C	Water	PCB-83	10	9	1	1	0	100	0	100
EPA 1668C	Water	PCB-84/92	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-85/116	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-86	10	4	6	5	1	83	17	90
EPA 1668C	Water	PCB-87/117/125	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-88/91	10	0	10	8	2	80	20	80

Table 5-3

Summary of Water Field Duplicate Results for Total PCBs by EPA Method 1668C in 2017

Method	Matrix	Analyte	Total Number Field Duplicate Pairs	Total Number Field Duplicate Pairs with NDs for Both Samples	Total Number Field Duplicate Pairs with Positives in Either Sample					Overall Percent Meet Criteria
					Total Number	Number Meet Criteria	Number Do Not Meet Criteria	Percent Meet Criteria	Percent Do Not Meet Criteria	
EPA 1668C	Water	PCB-89	10	3	7	6	1	86	14	90
EPA 1668C	Water	PCB-90/101	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-93	10	10	0	0	0	NA	NA	100
EPA 1668C	Water	PCB-94	10	1	9	7	2	78	22	80
EPA 1668C	Water	PCB-95/98/102	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-96	10	1	9	7	2	78	22	80
EPA 1668C	Water	PCB-97	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-99	10	1	9	7	2	78	22	80
EPA 1668C	Water	PCB-100	10	2	8	6	2	75	25	80
EPA 1668C	Water	PCB-103	10	1	9	7	2	78	22	80
EPA 1668C	Water	PCB-104	10	6	4	4	0	100	0	100
EPA 1668C	Water	PCB-105	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-106/118	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-107/109	10	1	9	7	2	78	22	80
EPA 1668C	Water	PCB-108/112	10	1	9	7	2	78	22	80
EPA 1668C	Water	PCB-110	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-111/115	10	2	8	7	1	88	13	90
EPA 1668C	Water	PCB-113	10	3	7	7	0	100	0	100
EPA 1668C	Water	PCB-114	10	1	9	7	2	78	22	80
EPA 1668C	Water	PCB-119	10	1	9	7	2	78	22	80
EPA 1668C	Water	PCB-120	10	4	6	6	0	100	0	100
EPA 1668C	Water	PCB-121	10	10	0	0	0	NA	NA	100
EPA 1668C	Water	PCB-122	10	3	7	6	1	86	14	90
EPA 1668C	Water	PCB-123	10	3	7	6	1	86	14	90
EPA 1668C	Water	PCB-124	10	1	9	8	1	89	11	90
EPA 1668C	Water	PCB-126	10	5	5	5	0	100	0	100
EPA 1668C	Water	PCB-127	10	10	0	0	0	NA	NA	100

Table 5-3

Summary of Water Field Duplicate Results for Total PCBs by EPA Method 1668C in 2017

Method	Matrix	Analyte	Total Number Field Duplicate Pairs	Total Number Field Duplicate Pairs with NDs for Both Samples	Total Number Field Duplicate Pairs with Positives in Either Sample					Overall Percent Meet Criteria
					Total Number	Number Meet Criteria	Number Do Not Meet Criteria	Percent Meet Criteria	Percent Do Not Meet Criteria	
EPA 1668C	Water	PCB-128/162	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-129	10	2	8	7	1	88	13	90
EPA 1668C	Water	PCB-130	10	1	9	7	2	78	22	80
EPA 1668C	Water	PCB-131/133	10	1	9	8	1	89	11	90
EPA 1668C	Water	PCB-132/161	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-142	10	0	10	10	0	100	0	100
EPA 1668C	Water	PCB-134/143	10	1	9	8	1	89	11	90
EPA 1668C	Water	PCB-135	10	1	9	6	3	67	33	70
EPA 1668C	Water	PCB-136	10	1	9	7	2	78	22	80
EPA 1668C	Water	PCB-137	10	2	8	7	1	88	13	90
EPA 1668C	Water	PCB-138/163/164	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-139/149	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-140	10	6	4	4	0	100	0	100
EPA 1668C	Water	PCB-141	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-142	10	10	0	0	0	NA	NA	100
EPA 1668C	Water	PCB-144	10	2	8	7	1	88	13	90
EPA 1668C	Water	PCB-145	10	10	0	0	0	NA	NA	100
EPA 1668C	Water	PCB-146/165	10	1	9	7	2	78	22	80
EPA 1668C	Water	PCB-147	10	4	6	4	2	67	33	80
EPA 1668C	Water	PCB-148	10	6	4	4	0	100	0	100
EPA 1668C	Water	PCB-150	10	6	4	4	0	100	0	100
EPA 1668C	Water	PCB-151	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-152	10	6	4	4	0	100	0	100
EPA 1668C	Water	PCB-153	10	0	10	9	1	90	10	90
EPA 1668C	Water	PCB-154	10	2	8	7	1	88	13	90
EPA 1668C	Water	PCB-155	10	10	0	0	0	NA	NA	100
EPA 1668C	Water	PCB-156	10	0	10	8	2	80	20	80

Table 5-3

Summary of Water Field Duplicate Results for Total PCBs by EPA Method 1668C in 2017

Method	Matrix	Analyte	Total Number Field Duplicate Pairs	Total Number Field Duplicate Pairs with NDs for Both Samples	Total Number Field Duplicate Pairs with Positives in Either Sample					Overall Percent Meet Criteria
					Total Number	Number Meet Criteria	Number Do Not Meet Criteria	Percent Meet Criteria	Percent Do Not Meet Criteria	
EPA 1668C	Water	PCB-157	10	1	9	8	1	89	11	90
EPA 1668C	Water	PCB-158/160	10	1	9	8	1	89	11	90
EPA 1668C	Water	PCB-159	10	7	3	3	0	100	0	100
EPA 1668C	Water	PCB-166	10	8	2	2	0	100	0	100
EPA 1668C	Water	PCB-167	10	1	9	8	1	89	11	90
EPA 1668C	Water	PCB-168	10	7	3	3	0	100	0	100
EPA 1668C	Water	PCB-169	10	10	0	0	0	NA	NA	100
EPA 1668C	Water	PCB-170	10	1	9	7	2	78	22	80
EPA 1668C	Water	PCB-171	10	1	9	7	2	78	22	80
EPA 1668C	Water	PCB-172	10	2	8	7	1	88	13	90
EPA 1668C	Water	PCB-173	10	9	1	1	0	100	0	100
EPA 1668C	Water	PCB-174	10	1	9	8	1	89	11	90
EPA 1668C	Water	PCB-175	10	7	3	3	0	100	0	100
EPA 1668C	Water	PCB-176	10	3	7	6	1	86	14	90
EPA 1668C	Water	PCB-177	10	1	9	7	2	78	22	80
EPA 1668C	Water	PCB-178	10	1	9	7	2	78	22	80
EPA 1668C	Water	PCB-179	10	3	7	5	2	71	29	80
EPA 1668C	Water	PCB-180	10	0	10	9	1	90	10	90
EPA 1668C	Water	PCB-181	10	9	1	1	0	100	0	100
EPA 1668C	Water	PCB-182/187	10	0	10	8	2	80	20	80
EPA 1668C	Water	PCB-183	10	1	9	7	2	78	22	80
EPA 1668C	Water	PCB-184	10	10	0	0	0	NA	NA	100
EPA 1668C	Water	PCB-185	10	5	5	4	1	80	20	90
EPA 1668C	Water	PCB-186	10	10	0	0	0	NA	NA	100
EPA 1668C	Water	PCB-188	10	9	1	1	0	100	0	100
EPA 1668C	Water	PCB-189	10	5	5	5	0	100	0	100
EPA 1668C	Water	PCB-190	10	1	9	8	1	89	11	90

Table 5-3

Summary of Water Field Duplicate Results for Total PCBs by EPA Method 1668C in 2017

Method	Matrix	Analyte	Total Number Field Duplicate Pairs	Total Number Field Duplicate Pairs with NDs for Both Samples	Total Number Field Duplicate Pairs with Positives in Either Sample					Overall Percent Meet Criteria
					Total Number	Number Meet Criteria	Number Do Not Meet Criteria	Percent Meet Criteria	Percent Do Not Meet Criteria	
EPA 1668C	Water	PCB-191	10	5	5	5	0	100	0	100
EPA 1668C	Water	PCB-192	10	10	0	0	0	NA	NA	100
EPA 1668C	Water	PCB-193	10	4	6	5	1	83	17	90
EPA 1668C	Water	PCB-194	10	1	9	7	2	78	22	80
EPA 1668C	Water	PCB-195	10	2	8	7	1	88	13	90
EPA 1668C	Water	PCB-196/203	10	1	9	8	1	89	11	90
EPA 1668C	Water	PCB-197	10	7	3	3	0	100	0	100
EPA 1668C	Water	PCB-198	10	6	4	4	0	100	0	100
EPA 1668C	Water	PCB-199	10	1	9	6	3	67	33	70
EPA 1668C	Water	PCB-200	10	4	6	5	1	83	17	90
EPA 1668C	Water	PCB-201	10	5	5	4	1	80	20	90
EPA 1668C	Water	PCB-202	10	3	7	6	1	86	14	90
EPA 1668C	Water	PCB-204	10	10	0	0	0	NA	NA	100
EPA 1668C	Water	PCB-205	10	5	5	5	0	100	0	100
EPA 1668C	Water	PCB-206	10	1	9	6	3	67	33	70
EPA 1668C	Water	PCB-207	10	5	5	4	1	80	20	90
EPA 1668C	Water	PCB-208	10	1	9	7	2	78	22	80
EPA 1668C	Water	PCB-209	10	1	9	8	1	89	11	90
EPA 1668C	Water	All results[1]	1800	449	1351	1092	259	81	19	86

Notes:

1 . All results = total number field duplicate pairs multiplied by the number of analytes determined by the method.

ND: not detected

PCB: polychlorinated biphenyl

RAMP: Remedial Action Monitoring Program

Table 5-4

Summary of Water Field Duplicate Results for TSS in 2017

Method	Analyte	Total Number Field Duplicate Pairs	Total Number Field Duplicate Pairs with NDs for Both Samples	Total Number Field Duplicate Pairs with Positives in Either Sample					Overall Percent Meet Criteria
				Total Number	Number Meet Criteria	Number Do Not Meet Criteria	Percent Meet Criteria	Percent Do Not Meet Criteria	
SM 2540D	Total suspended solids	9	1	8	7	1	88	13	89

Notes:

All results = total number field duplicate pairs multiplied by the number of analytes determined by the method.

ND: not detected

PCB: polychlorinated biphenyl

Table 5-5

Summary of RAMP Fish Laboratory Duplicate Results for Aroclor PCBs in 2017

Method	Analyte	Total Number Laboratory Duplicate Pairs	Total Number Laboratory Duplicate Pairs with NDs for Both Samples	Total Number Laboratory Duplicate Pairs with Positives in Either Sample					Overall Percent Meet Criteria
				Total Number	Number Meet Criteria	Number Do Not Meet Criteria	Percent Meet Criteria	Percent Do Not Meet Criteria	
SW-846 8082A	Total PCB	56	1	55	55	0	100	0	100
SW-846 8082A	Aroclor 1016	56	56	0	0	0	NA	NA	100
SW-846 8082A	Aroclor 1221	56	42	14	14	0	100	0.0	100
SW-846 8082A	Aroclor 1232	56	56	0	0	0	NA	NA	100
SW-846 8082A	Aroclor 1242	56	56	0	0	0	NA	NA	100
SW-846 8082A	Aroclor 1248	56	2	54	54	0	100	0.0	100
SW-846 8082A	Aroclor 1254	56	3	53	53	0	100	0.0	100
SW-846 8082A	Aroclor 1260	56	5	51	51	0	100	0.0	100
SW-846 8082A	All results	448	221	227	227	0	100	0.0	100

Notes:

All results = total number field duplicate pairs multiplied by the number of analytes determined by the method.

ND: not detected

PCB: polychlorinated biphenyl

RAMP: Remedial Action Monitoring Program

Table 5-6

Summary of Fish Laboratory Duplicate Results for Lipids in 2017

Method	Analyte	Total Number Laboratory Duplicate Pairs	Total Number Laboratory Duplicate Pairs with NDs for Both Samples	Total Number Laboratory Duplicate Pairs with Positives in Either Sample					Overall Percent Meet Criteria
				Total Number	Number Meet Criteria	Number Do Not Meet Criteria	Percent Meet Criteria	Percent Do Not Meet Criteria	
S-GB-O-068-REV.01	Lipids	56	0	56	55	1	98	2	98

Notes:

All results = total number field duplicate pairs multiplied by the number of analytes determined by the method.

ND: not detected

Table 5-7

Summary Statistics of 2017 Equipment Blanks for Water Sampling Program

Analyte	Method	Number Equipment Blanks	Equipment Blanks with Results > MDL	Minimum Concentration	Maximum Concentration	Average Concentration	Median Concentration	Concentration Units	Percent Contaminated
Total PCBs	EPA 1668C	8	8	0.0400	0.344	0.137	0.114	ng/L	100%
Decachlorobiphenyl	EPA 1668C	8	2	0.000281	0.000717	0.000499	0.000499	ng/L	25%
Dichlorobiphenyl	EPA 1668C	8	4	0.0108	0.0507	0.0253	0.0199	ng/L	50%
Heptachlorobiphenyl	EPA 1668C	8	3	0.000553	0.00135	0.000910	0.000828	ng/L	38%
Hexachlorobiphenyl	EPA 1668C	8	7	0.00219	0.0214	0.00680	0.00353	ng/L	88%
Monochlorobiphenyl	EPA 1668C	8	6	0.00226	0.00668	0.00474	0.00496	ng/L	75%
Nonachlorobiphenyl	EPA 1668C	8	1	0.00132	0.00132	0.00132	0.00132	ng/L	13%
Octachlorobiphenyl	EPA 1668C	8	4	0.000639	0.00105	0.000874	0.000904	ng/L	50%
Pentachlorobiphenyl	EPA 1668C	8	7	0.00657	0.0203	0.0133	0.0117	ng/L	88%
Tetrachlorobiphenyl	EPA 1668C	8	8	0.00633	0.102	0.0462	0.0426	ng/L	100%
Trichlorobiphenyl	EPA 1668C	8	8	0.0138	0.167	0.0561	0.0489	ng/L	100%
PCB-001	EPA 1668C	8	6	0.00226	0.00668	0.00459	0.00495	ng/L	75%
PCB-003	EPA 1668C	8	1	0.000869	0.000869	0.000869	0.000869	ng/L	13%
PCB-004/010	EPA 1668C	8	2	0.0107	0.0132	0.0120	0.0120	ng/L	25%
PCB-005/008	EPA 1668C	8	2	0.0107	0.0155	0.0131	0.0131	ng/L	25%
PCB-011	EPA 1668C	8	4	0.00802	0.0139	0.0108	0.0106	ng/L	50%
PCB-015	EPA 1668C	8	1	0.00805	0.00805	0.00805	0.00805	ng/L	13%
PCB-016/032	EPA 1668C	8	7	0.00329	0.0328	0.0102	0.00724	ng/L	88%
PCB-017	EPA 1668C	8	7	0.00201	0.0162	0.00549	0.00424	ng/L	88%
PCB-018	EPA 1668C	8	8	0.00379	0.0296	0.00968	0.00684	ng/L	100%
PCB-019	EPA 1668C	8	6	0.00185	0.00933	0.00457	0.00414	ng/L	75%
PCB-020/021/033	EPA 1668C	8	7	0.00301	0.0172	0.00764	0.00635	ng/L	88%
PCB-022	EPA 1668C	8	7	0.00185	0.0118	0.00460	0.00345	ng/L	88%
PCB-024/027	EPA 1668C	8	6	0.000814	0.00641	0.00269	0.00248	ng/L	75%
PCB-025	EPA 1668C	8	1	0.00102	0.00102	0.00102	0.00102	ng/L	13%
PCB-026	EPA 1668C	8	5	0.00085	0.00198	0.00156	0.00169	ng/L	63%
PCB-028	EPA 1668C	8	7	0.00331	0.0221	0.00847	0.00647	ng/L	88%
PCB-031	EPA 1668C	8	7	0.00284	0.0167	0.00722	0.00597	ng/L	88%
PCB-037	EPA 1668C	8	4	0.00158	0.00488	0.00331	0.00340	ng/L	50%
PCB-040	EPA 1668C	8	4	0.00182	0.00373	0.00240	0.00203	ng/L	50%
PCB-41/64/71/72	EPA 1668C	8	8	0.00238	0.0129	0.00649	0.00607	ng/L	100%

Table 5-7

Summary Statistics of 2017 Equipment Blanks for Water Sampling Program

Analyte	Method	Number Equipment Blanks	Equipment Blanks with Results > MDL	Minimum Concentration	Maximum Concentration	Average Concentration	Median Concentration	Concentration Units	Percent Contaminated
PCB-042/059	EPA 1668C	8	5	0.00217	0.00630	0.00327	0.00267	ng/L	63%
PCB-043/049	EPA 1668C	8	8	0.00234	0.0123	0.00488	0.00412	ng/L	100%
PCB-044	EPA 1668C	8	5	0.00495	0.0171	0.00853	0.00635	ng/L	63%
PCB-045	EPA 1668C	8	3	0.00142	0.00572	0.00374	0.00408	ng/L	38%
PCB-046	EPA 1668C	8	4	0.000897	0.00301	0.00190	0.00185	ng/L	50%
PCB-047	EPA 1668C	8	7	0.00279	0.0132	0.00612	0.00370	ng/L	88%
PCB-048/075	EPA 1668C	8	5	0.000769	0.00161	0.00116	0.00128	ng/L	63%
PCB-051	EPA 1668C	8	4	0.00128	0.00273	0.00204	0.00207	ng/L	50%
PCB-052/069	EPA 1668C	8	6	0.00418	0.0137	0.00681	0.00598	ng/L	75%
PCB-053	EPA 1668C	8	5	0.000889	0.00500	0.00234	0.00191	ng/L	63%
PCB-056/060	EPA 1668C	8	7	0.00202	0.00472	0.00336	0.00353	ng/L	88%
PCB-061/070	EPA 1668C	8	8	0.00161	0.00702	0.00399	0.00381	ng/L	100%
PCB-066/076	EPA 1668C	8	4	0.00237	0.00508	0.00356	0.00339	ng/L	50%
PCB-068	EPA 1668C	8	2	0.000950	0.00210	0.00153	0.00153	ng/L	25%
PCB-074	EPA 1668C	8	6	0.00104	0.00184	0.00134	0.00123	ng/L	75%
PCB-077	EPA 1668C	8	1	0.00110	0.00110	0.00110	0.00110	ng/L	13%
PCB-084/092	EPA 1668C	8	4	0.00135	0.00256	0.00207	0.00218	ng/L	50%
PCB-085/116	EPA 1668C	8	2	0.000723	0.000921	0.000822	0.000822	ng/L	25%
PCB-087/117/125	EPA 1668C	8	4	0.00111	0.00142	0.00120	0.00113	ng/L	50%
PCB-088/091	EPA 1668C	8	1	0.00147	0.00147	0.00147	0.00147	ng/L	13%
PCB-090/101	EPA 1668C	8	3	0.00287	0.00439	0.00350	0.00324	ng/L	38%
PCB-095/098/102	EPA 1668C	8	5	0.00301	0.00518	0.00378	0.00314	ng/L	63%
PCB-097	EPA 1668C	8	5	0.000990	0.00144	0.00118	0.00114	ng/L	63%
PCB-099	EPA 1668C	8	5	0.000592	0.00180	0.00121	0.00120	ng/L	63%
PCB-105	EPA 1668C	8	3	0.00083	0.00170	0.00124	0.00118	ng/L	38%
PCB-106/118	EPA 1668C	8	5	0.00102	0.00286	0.00192	0.00175	ng/L	63%
PCB-110	EPA 1668C	8	7	0.00183	0.0041	0.00305	0.00296	ng/L	88%
PCB-126	EPA 1668C	8	1	0.000889	0.000889	0.000889	0.000889	ng/L	13%
PCB-128/162	EPA 1668C	8	1	0.00127	0.00127	0.00127	0.00127	ng/L	13%
PCB-132/161	EPA 1668C	8	2	0.00106	0.00167	0.00137	0.00137	ng/L	25%
PCB-136	EPA 1668C	8	2	0.000886	0.00111	0.000998	0.000998	ng/L	25%

Table 5-7

Summary Statistics of 2017 Equipment Blanks for Water Sampling Program

Analyte	Method	Number Equipment Blanks	Equipment Blanks with Results > MDL	Minimum Concentration	Maximum Concentration	Average Concentration	Median Concentration	Concentration Units	Percent Contaminated
PCB-138/163/164	EPA 1668C	8	3	0.00130	0.00362	0.00216	0.00155	ng/L	38%
PCB-139/149	EPA 1668C	8	6	0.00178	0.00469	0.00293	0.00252	ng/L	75%
PCB-141	EPA 1668C	8	1	0.000996	0.000996	0.000996	0.000996	ng/L	13%
PCB-151	EPA 1668C	8	2	0.00119	0.00163	0.00141	0.00141	ng/L	25%
PCB-153	EPA 1668C	8	6	0.000407	0.00384	0.00182	0.00168	ng/L	75%
PCB-156	EPA 1668C	8	1	0.000826	0.000826	0.000826	0.000826	ng/L	13%
PCB-158/160	EPA 1668C	8	1	0.000910	0.000910	0.000910	0.000910	ng/L	13%
PCB-169	EPA 1668C	8	1	0.00111	0.00111	0.00111	0.00111	ng/L	13%
PCB-179	EPA 1668C	8	1	0.000689	0.000689	0.000689	0.000689	ng/L	13%
PCB-180	EPA 1668C	8	1	0.000828	0.000828	0.000828	0.000828	ng/L	13%
PCB-184	EPA 1668C	8	1	0.000553	0.000553	0.000553	0.000553	ng/L	13%
PCB-193	EPA 1668C	8	1	0.000657	0.000657	0.000657	0.000657	ng/L	13%
PCB-194	EPA 1668C	8	3	0.000639	0.00105	0.000889	0.000978	ng/L	38%
PCB-196/203	EPA 1668C	8	1	0.000829	0.000829	0.000829	0.000829	ng/L	13%
PCB-206	EPA 1668C	8	1	0.000728	0.000728	0.000728	0.000728	ng/L	13%
PCB-207	EPA 1668C	8	1	0.000593	0.000593	0.000593	0.000593	ng/L	13%
PCB-209	EPA 1668C	8	2	0.000281	0.000717	0.000499	0.000499	ng/L	25%

Notes:

ng/L: nanograms per liter

MDL: method detection limit

RAMP: Remedial Action Monitoring Program

Figures

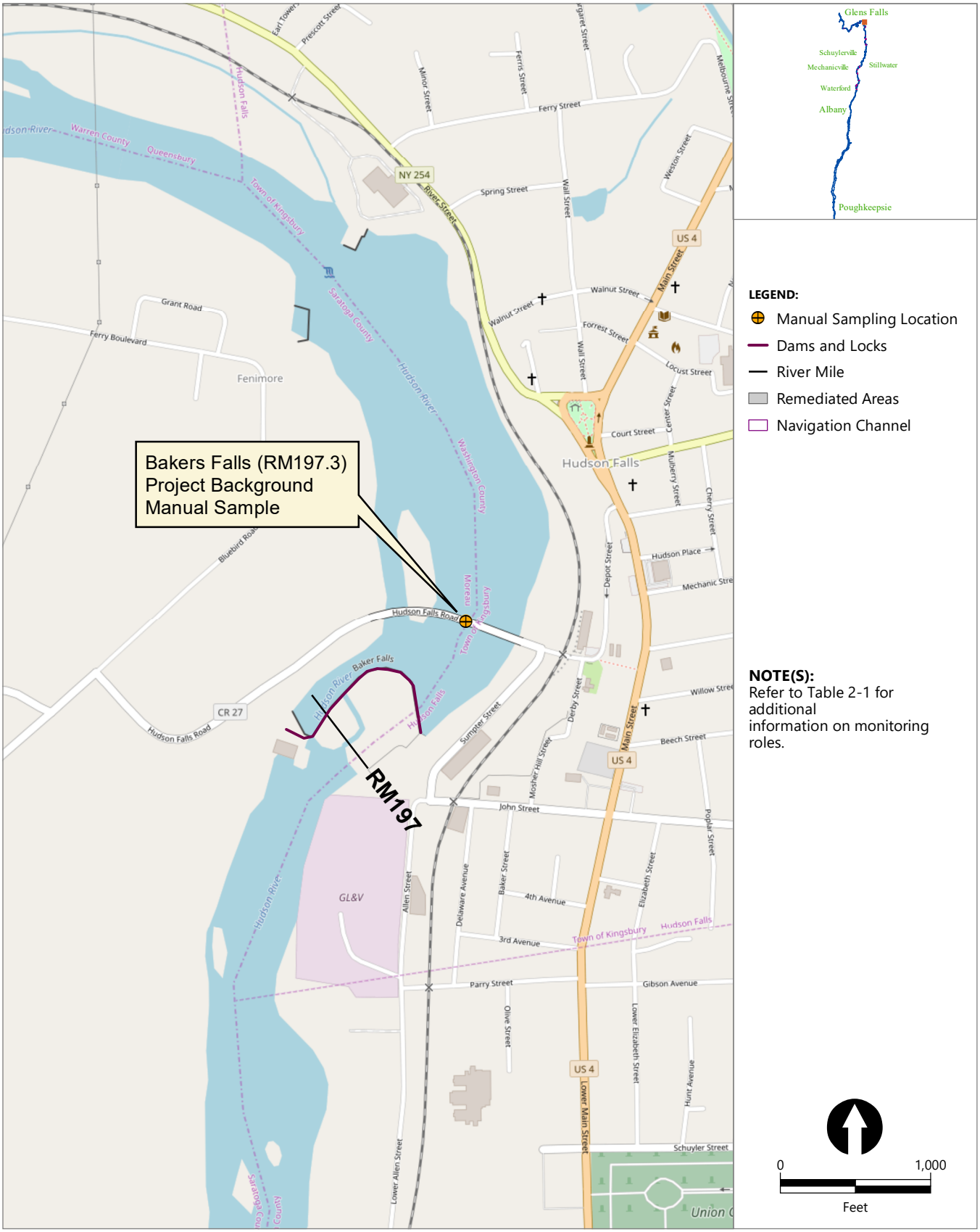

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Figure 2-1a
Water Monitoring Locations
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company

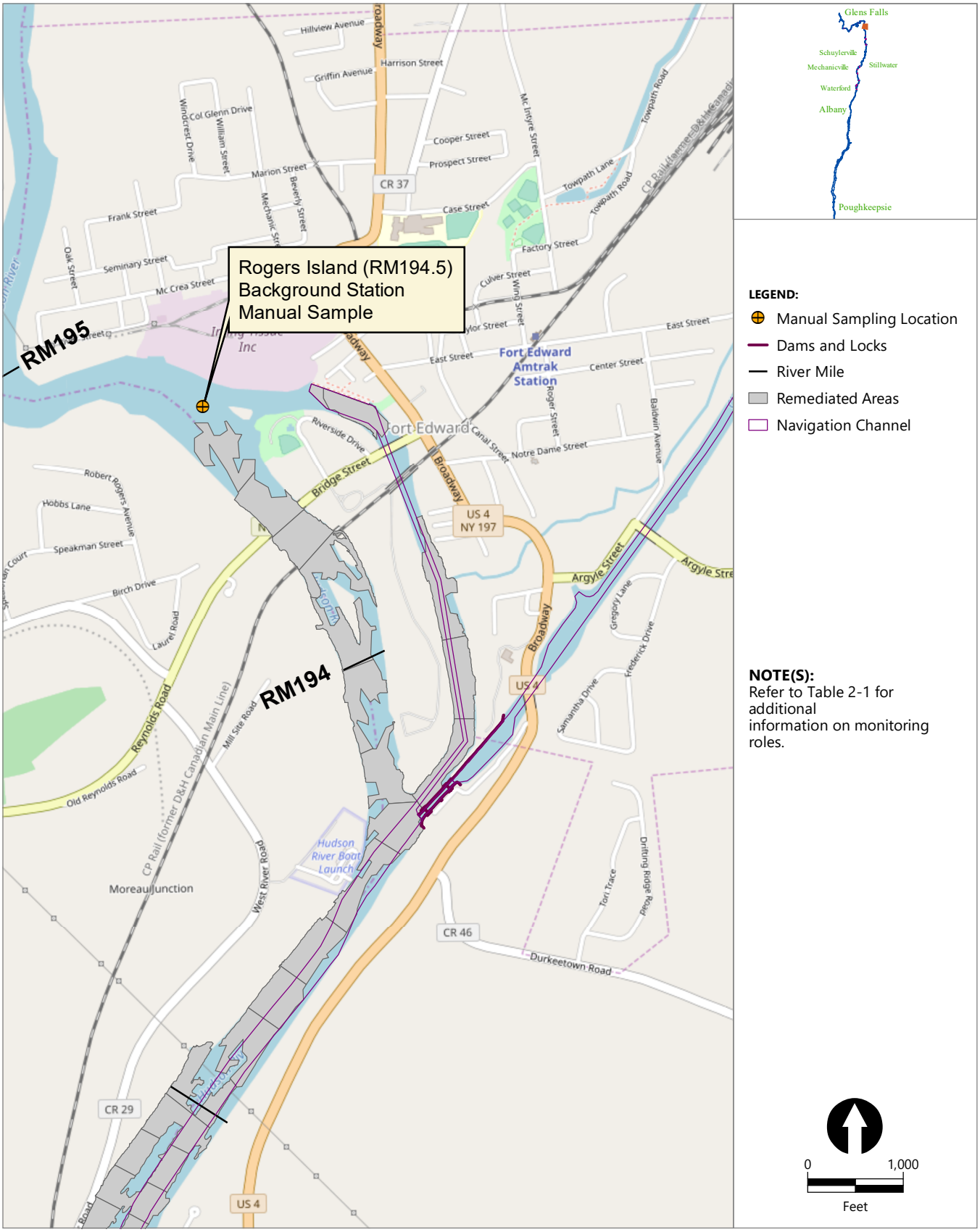

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Figure 2-1b
Water Monitoring Locations
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company

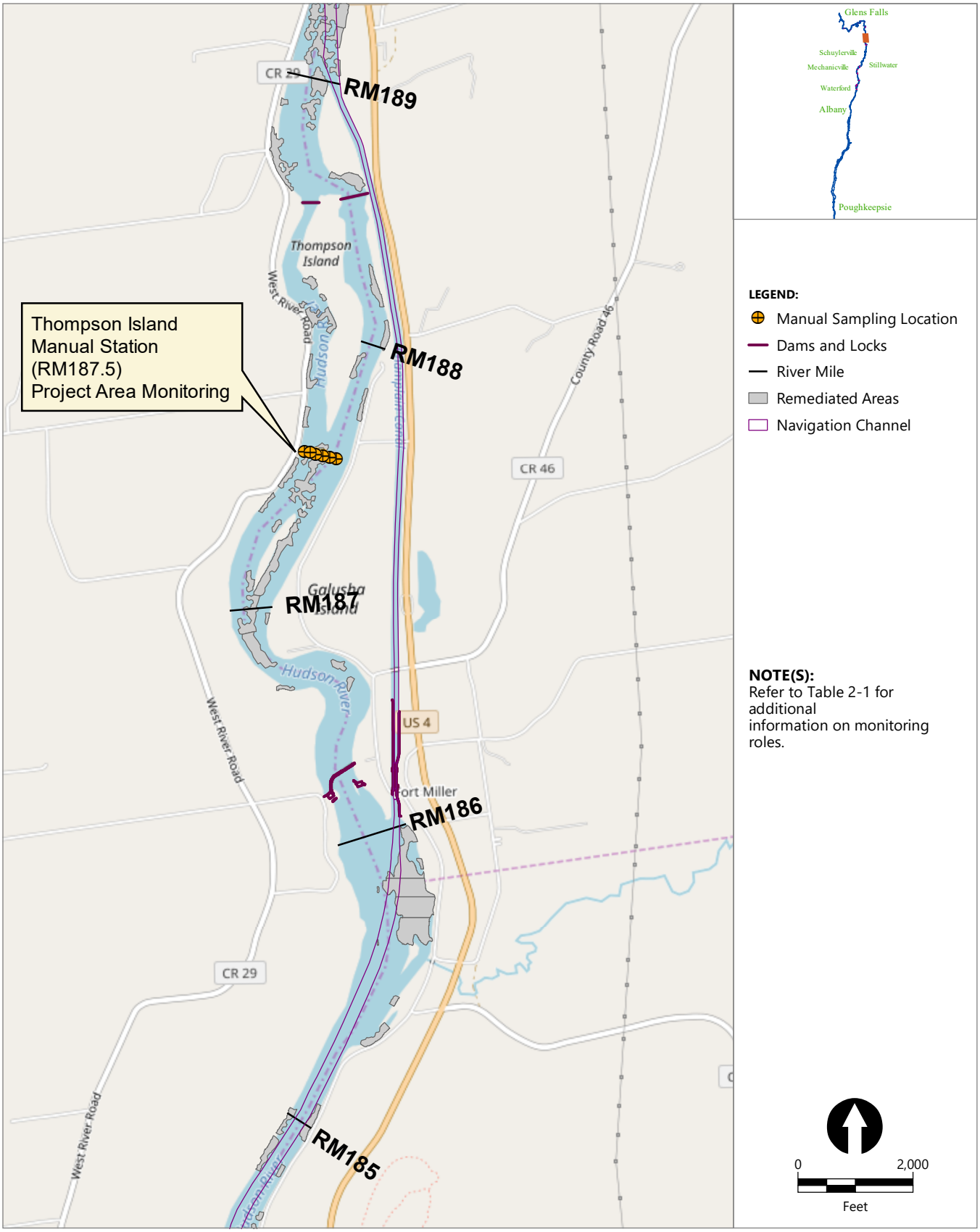

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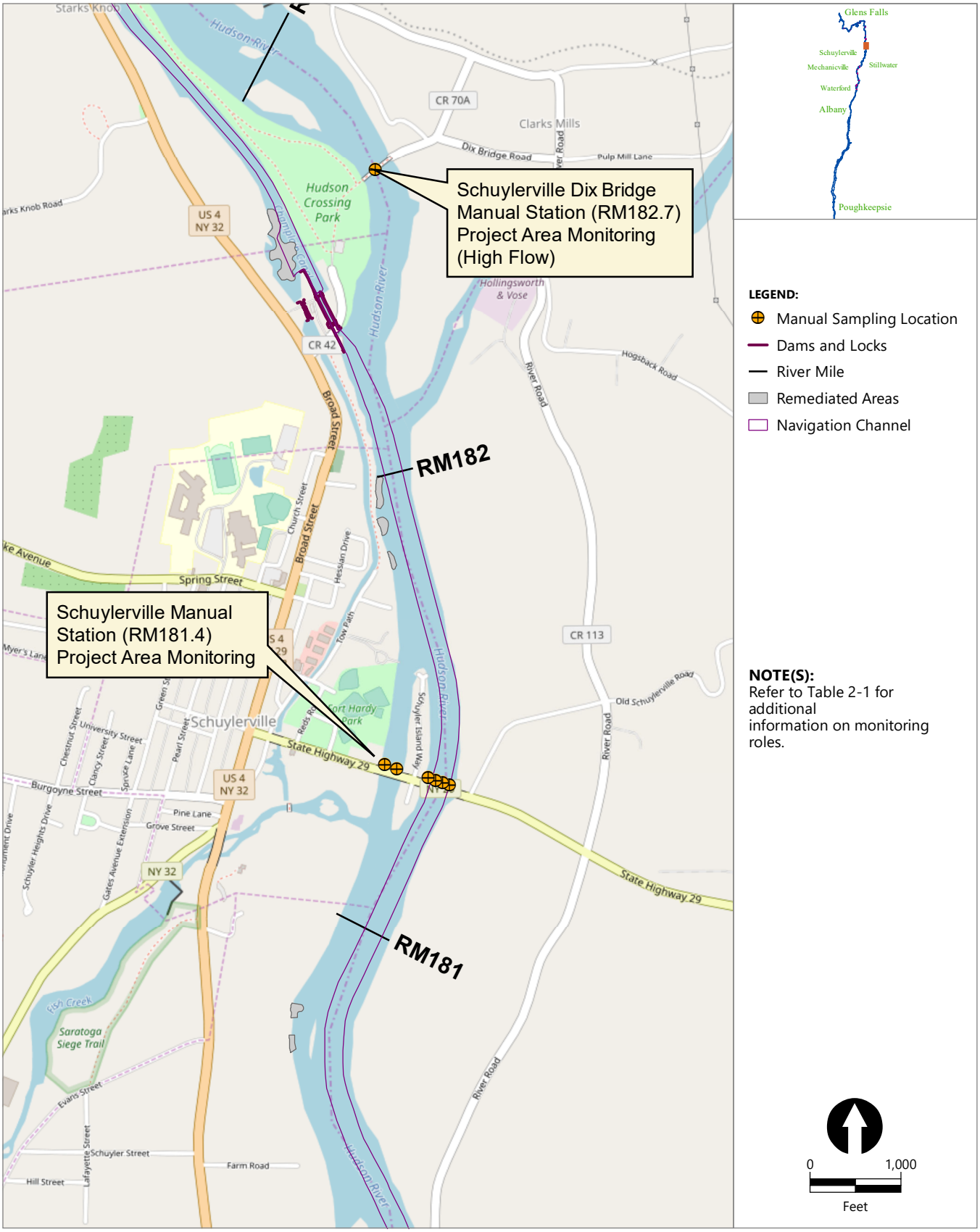
Figure 2-1c
Water Monitoring Locations
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company



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ANCHOR QEA

Figure 2-1d
Water Monitoring Locations
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company

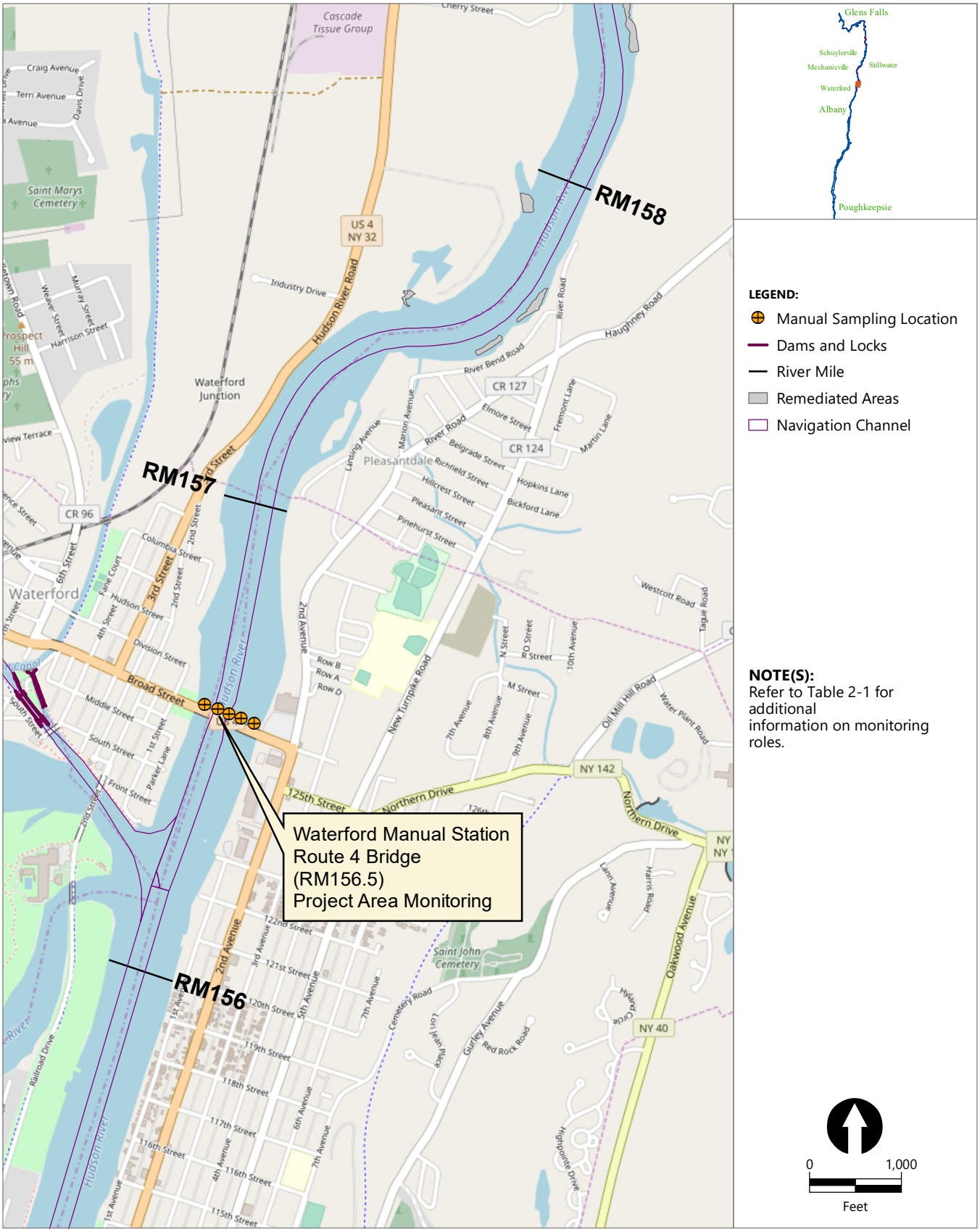

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Figure 2-1e
Water Monitoring Locations
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company

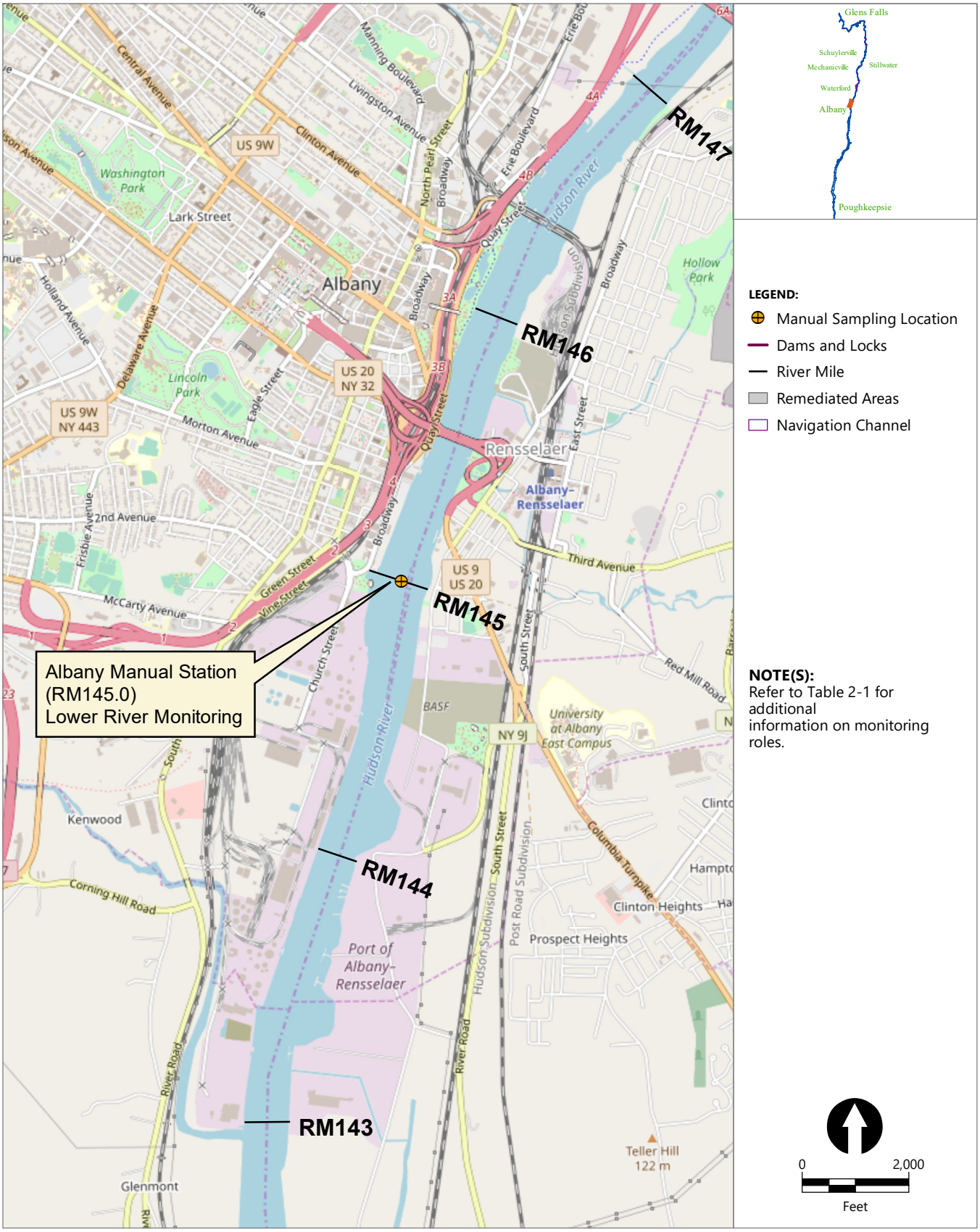

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Figure 2-1f
Water Monitoring Locations
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company

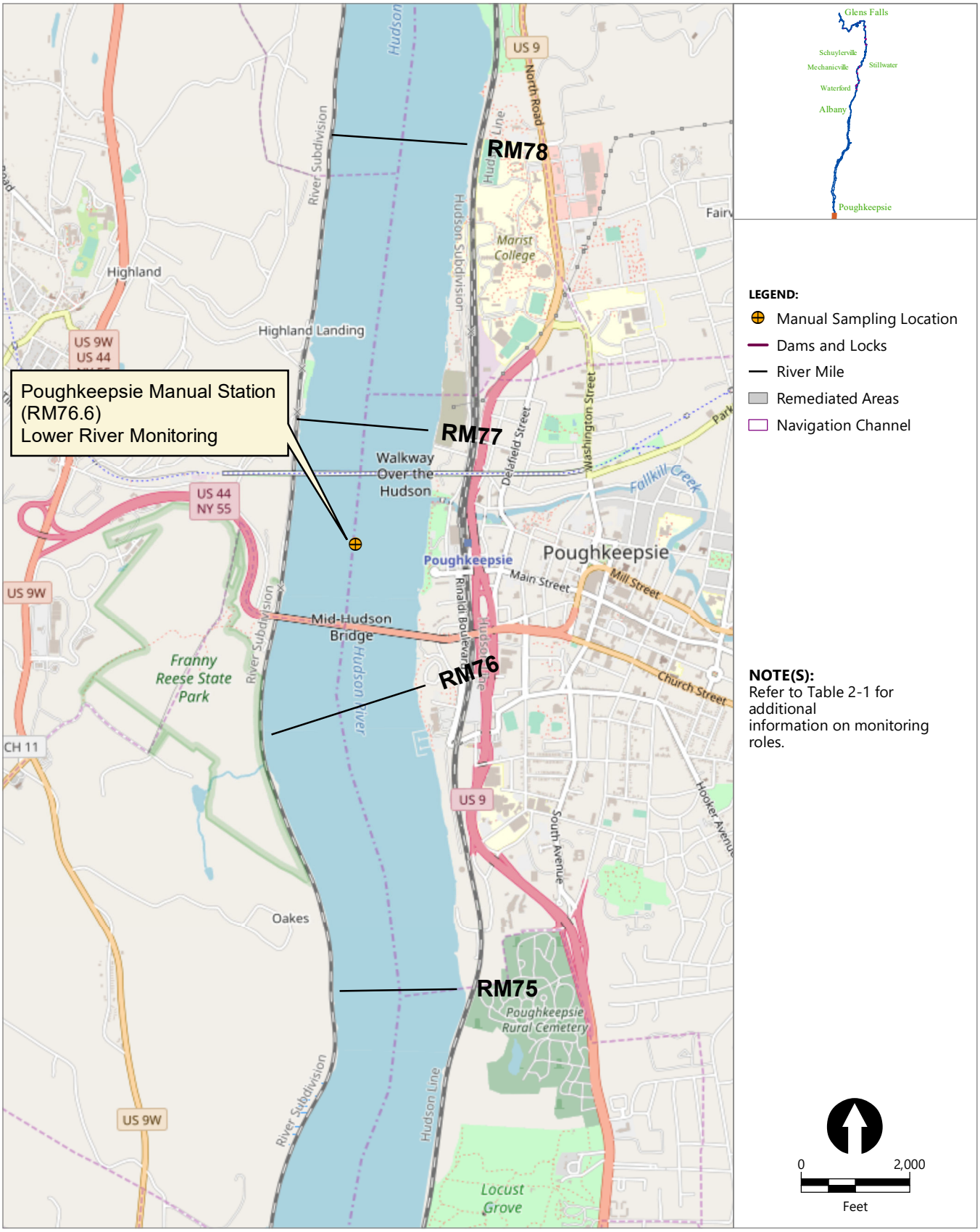

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Figure 2-1g
Water Monitoring Locations
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company

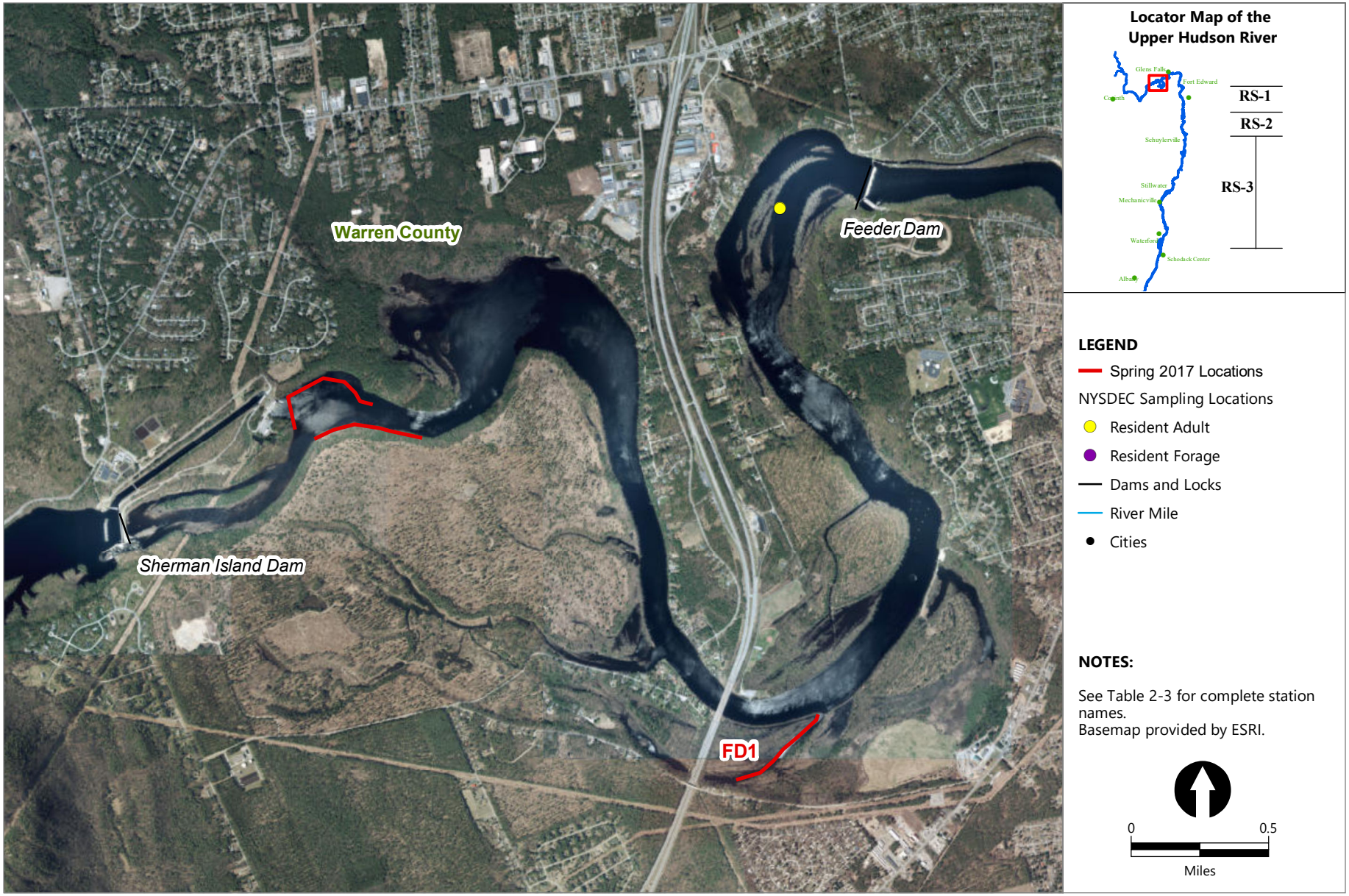

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Figure 2-2a
Spring 2017 Fish Sampling Locations
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company

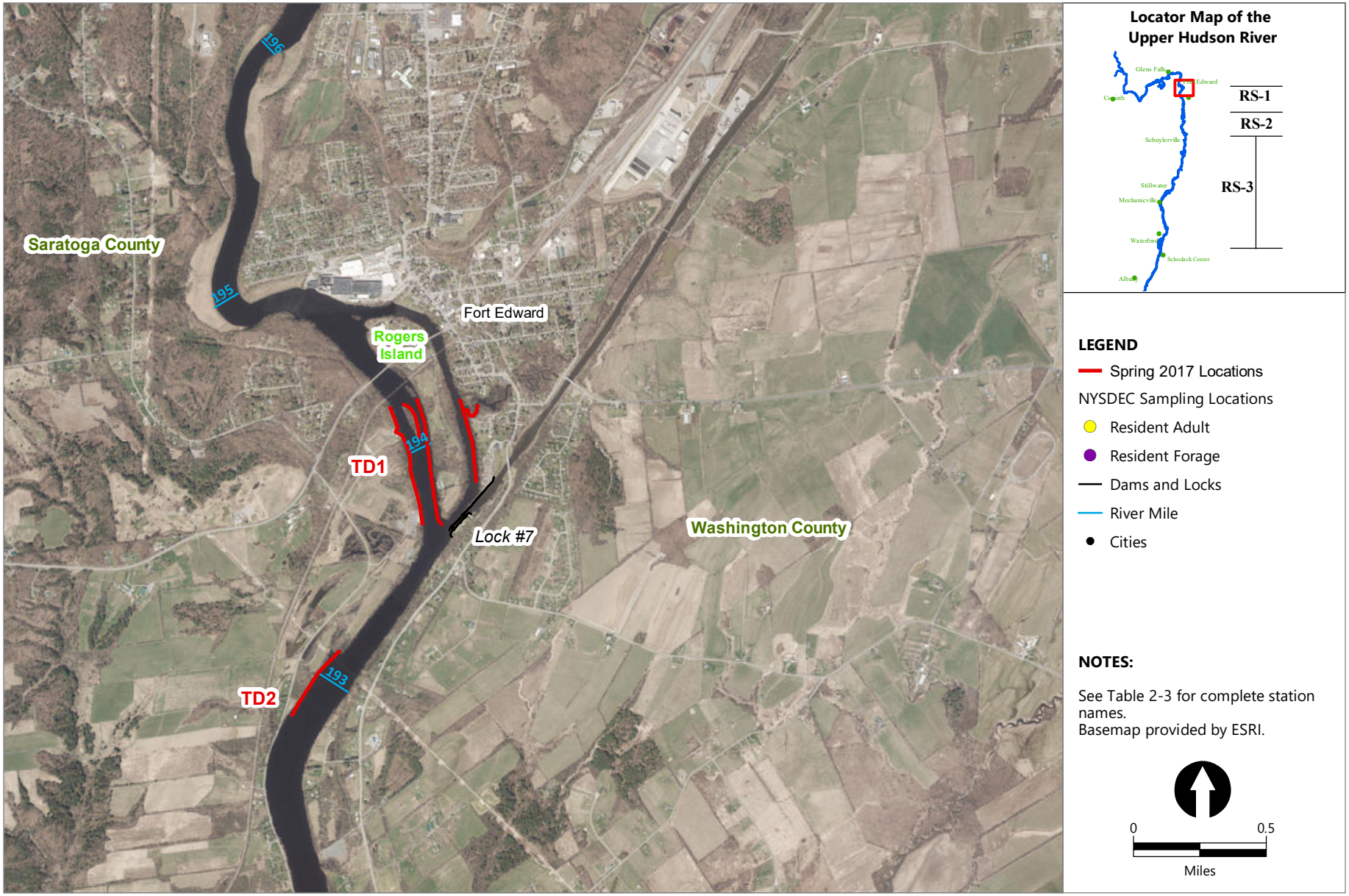

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Figure 2-2b
Spring 2017 Fish Sampling Locations
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company

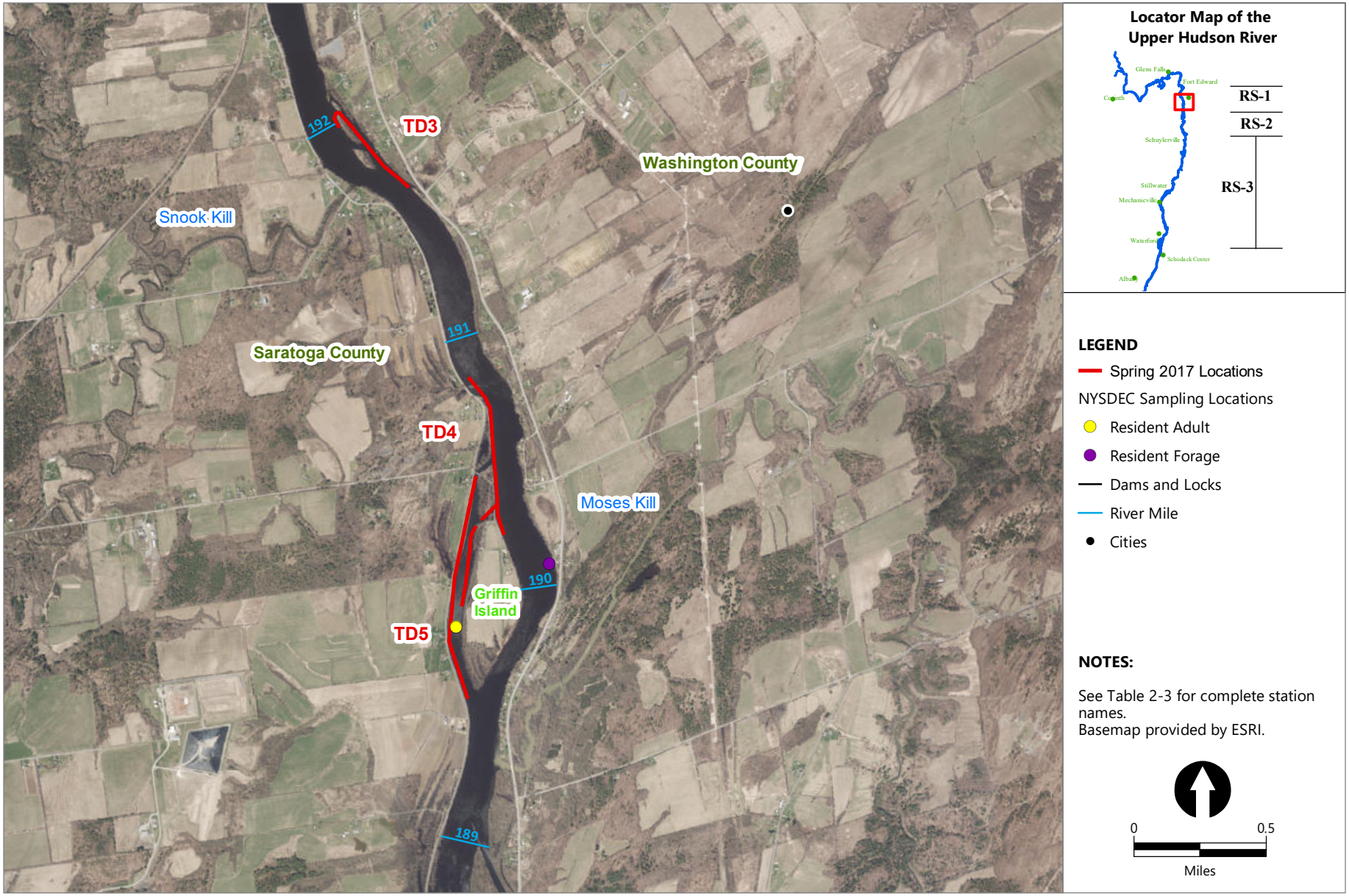

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Figure 2-2c
Spring 2017 Fish Sampling Locations
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company

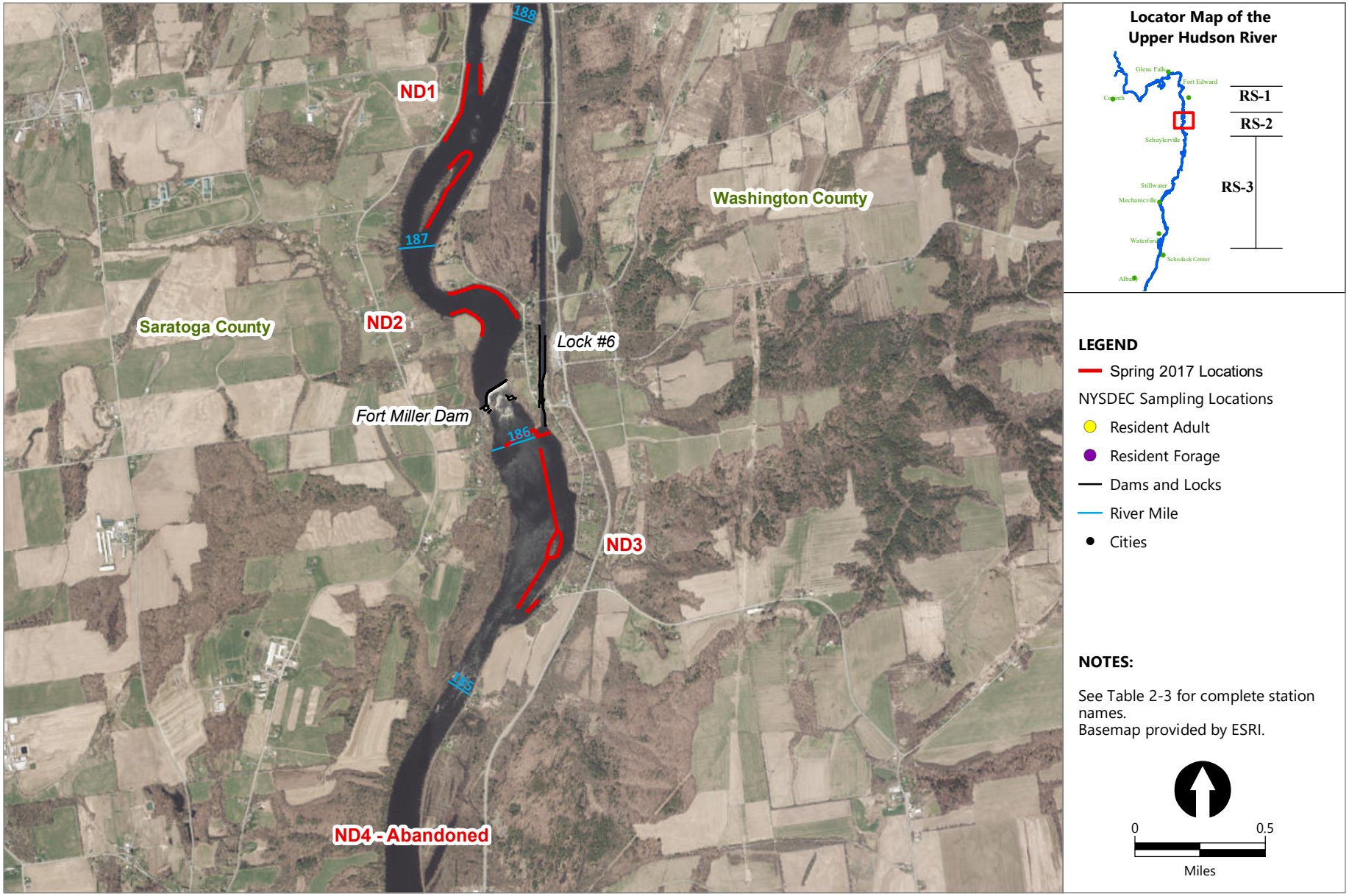

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Figure 2-2d
Spring 2017 Fish Sampling Locations
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company

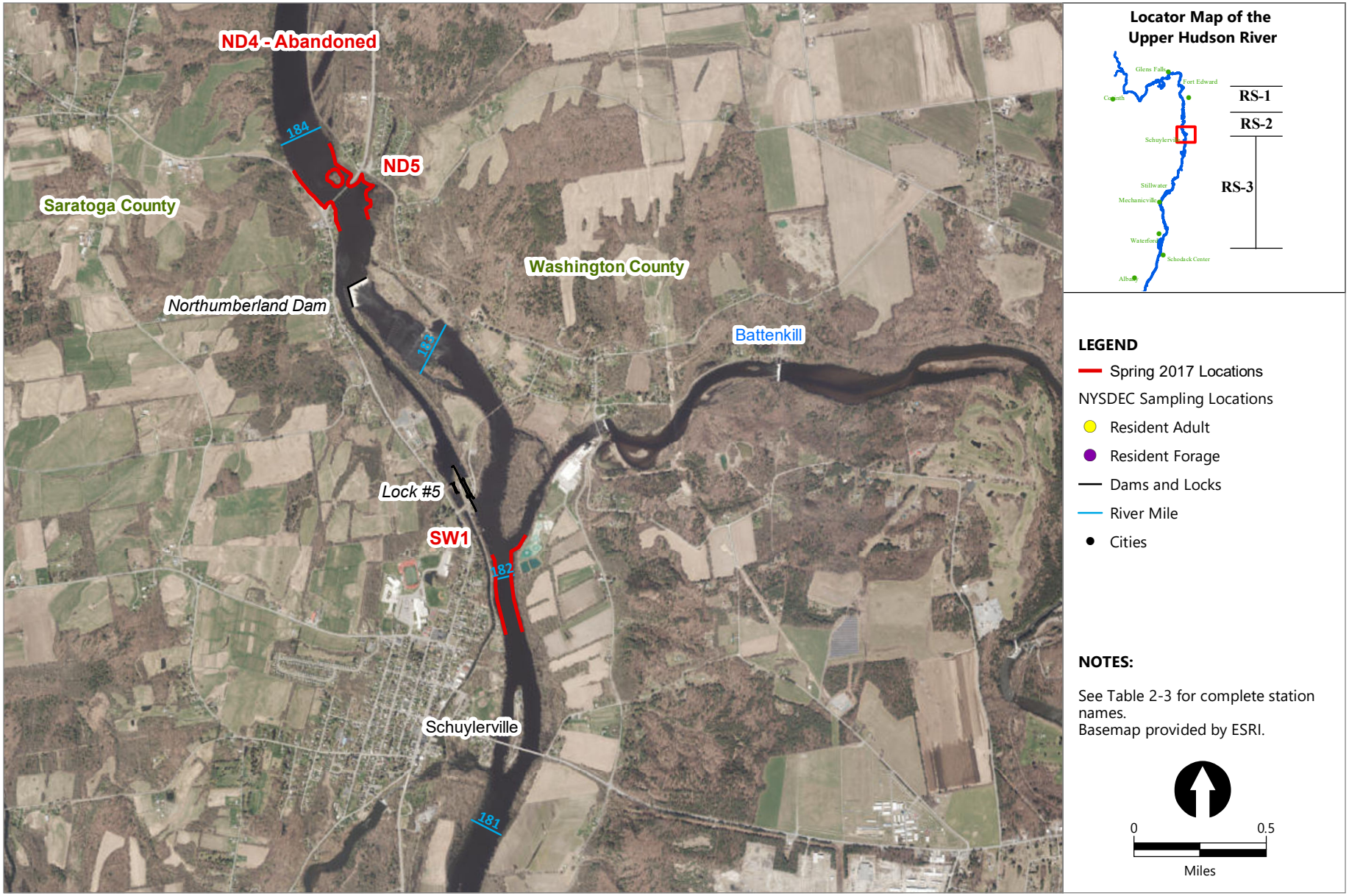

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Figure 2-2e
Spring 2017 Fish Sampling Locations
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company

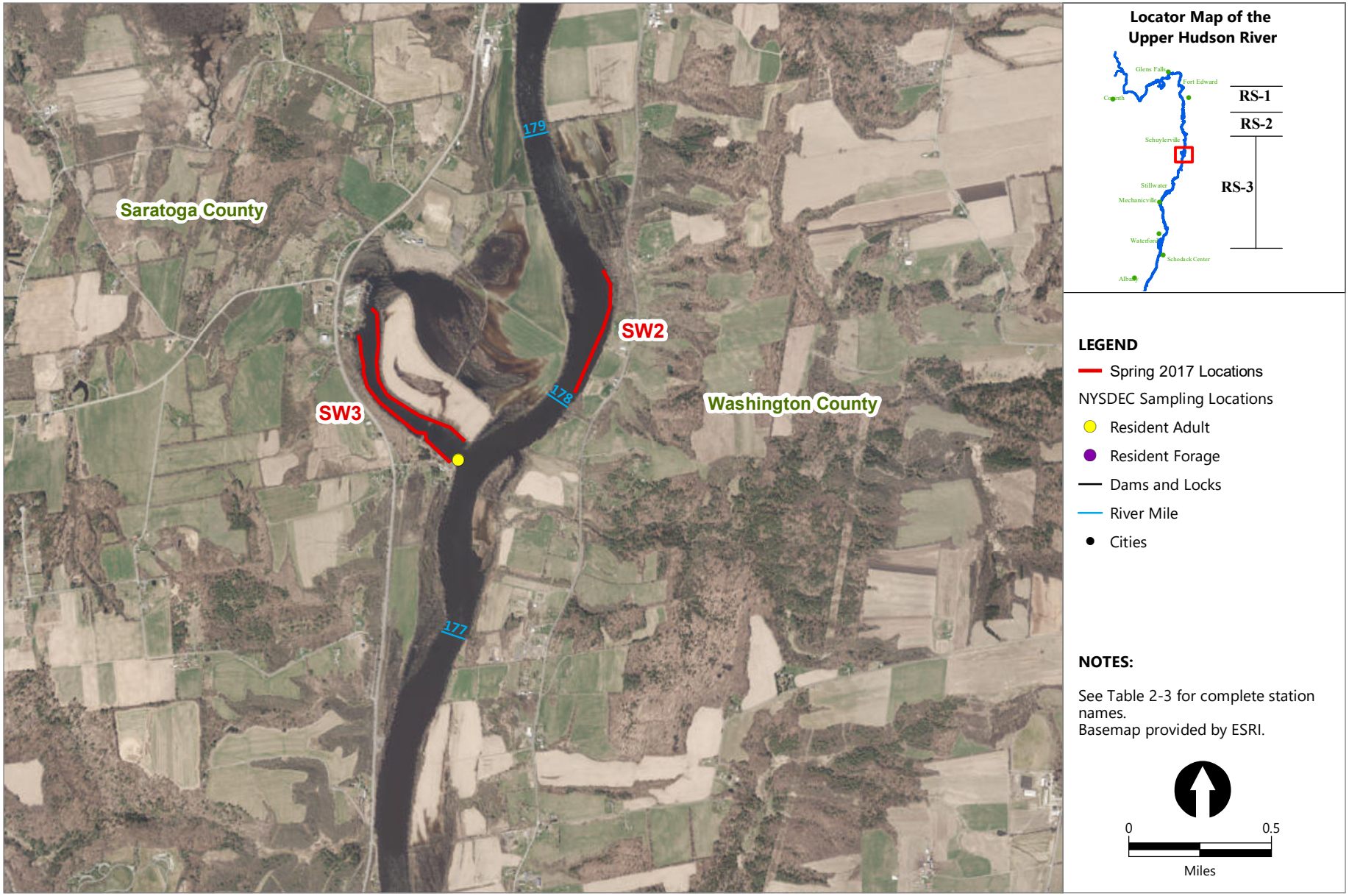

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Figure 2-2f
Spring 2017 Fish Sampling Locations
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company

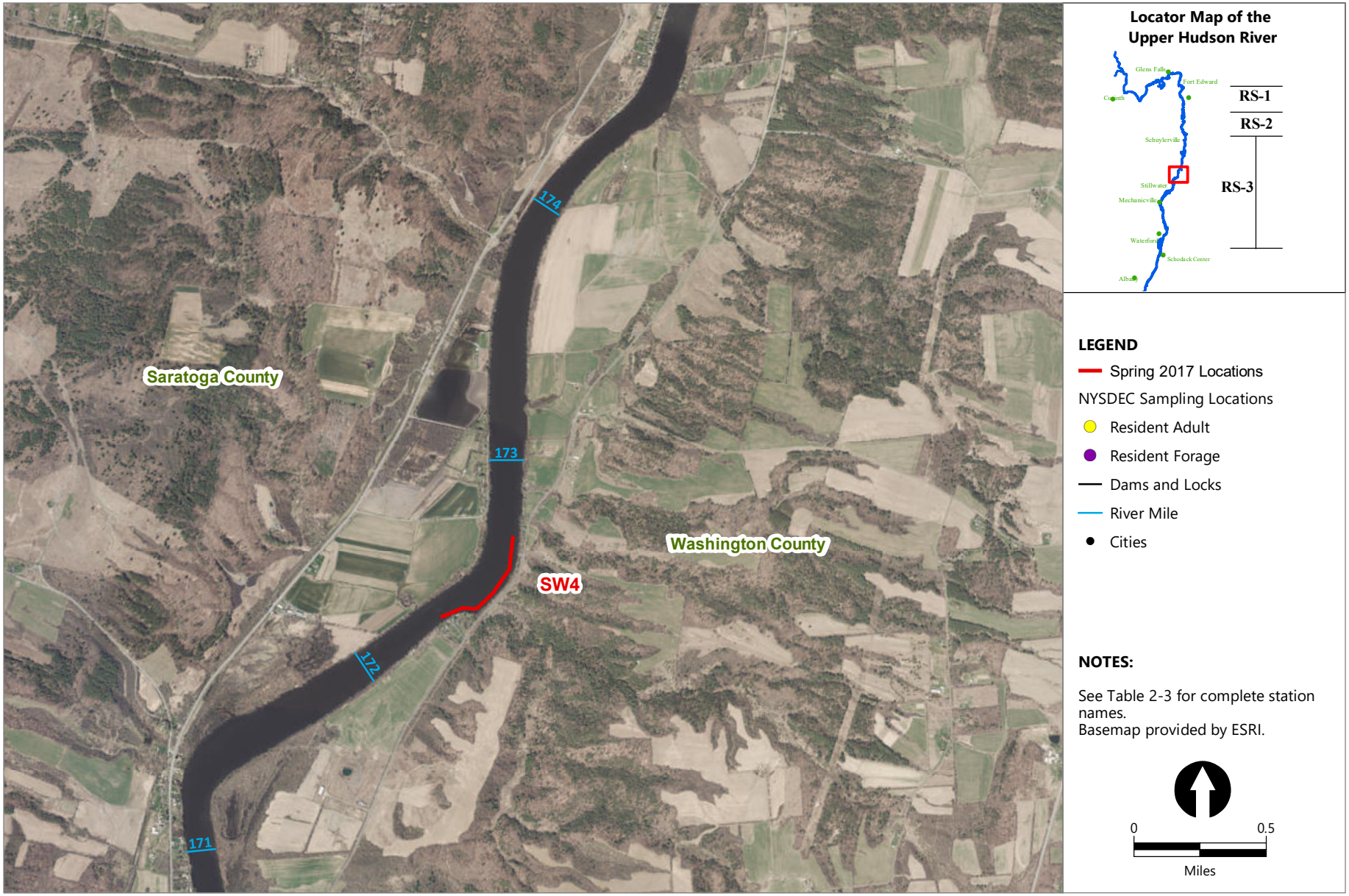

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Figure 2-2g
Spring 2017 Fish Sampling Locations
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company

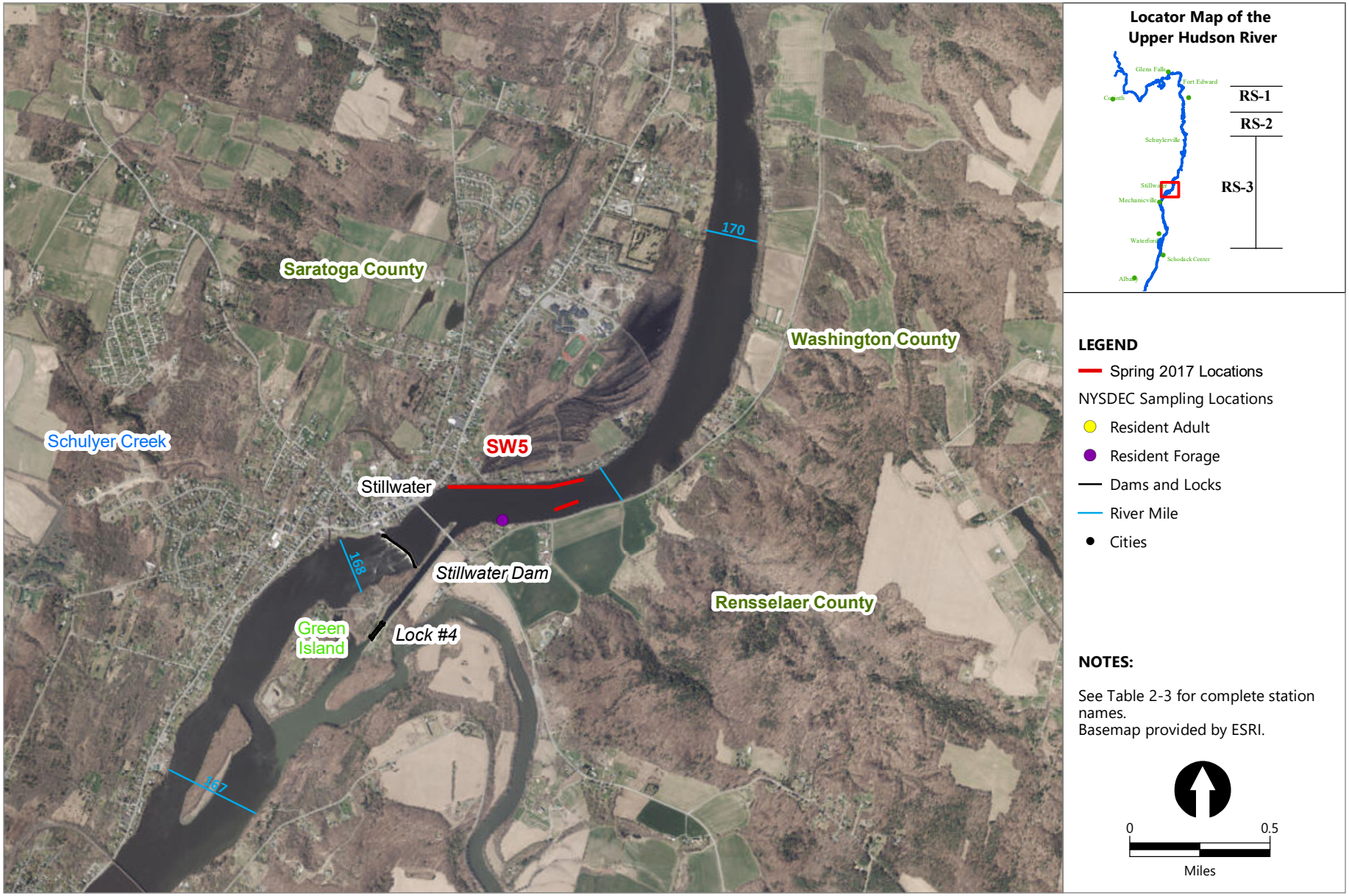

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Figure 2-2h
Spring 2017 Fish Sampling Locations
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company

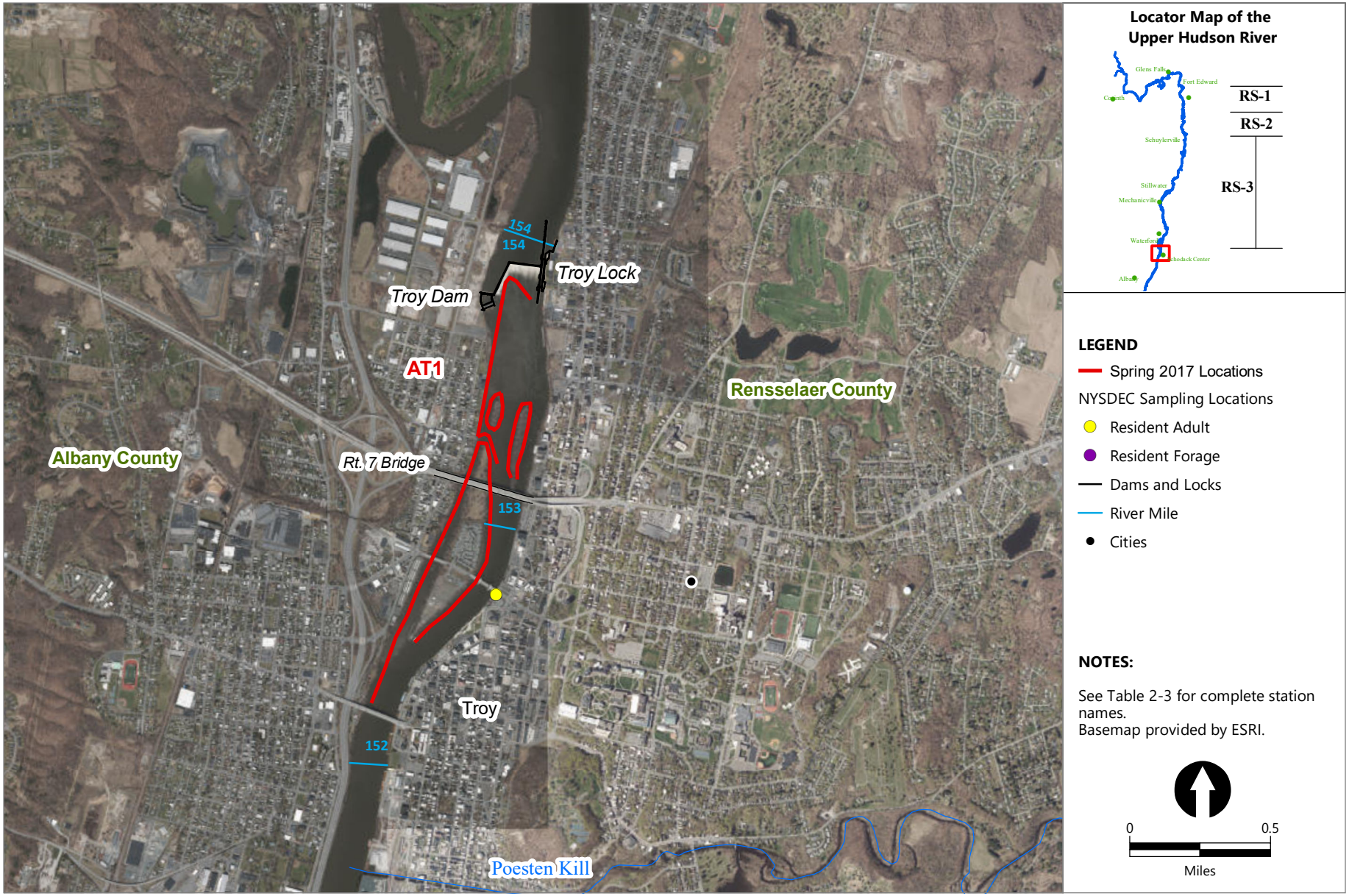

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Figure 2-2i
Spring 2017 Fish Sampling Locations
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company

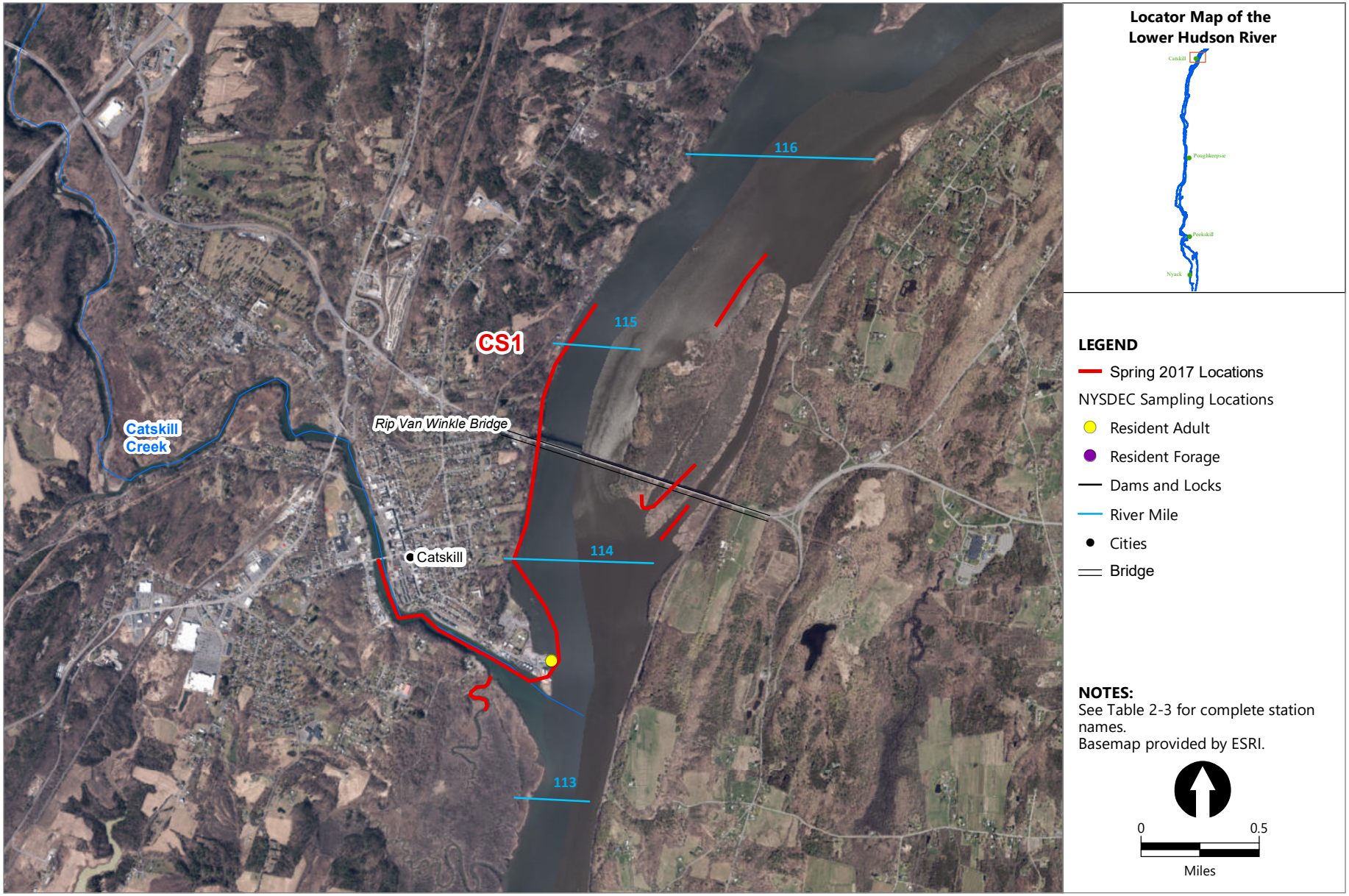

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Figure 2-2j
Spring 2017 Fish Sampling Locations
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company

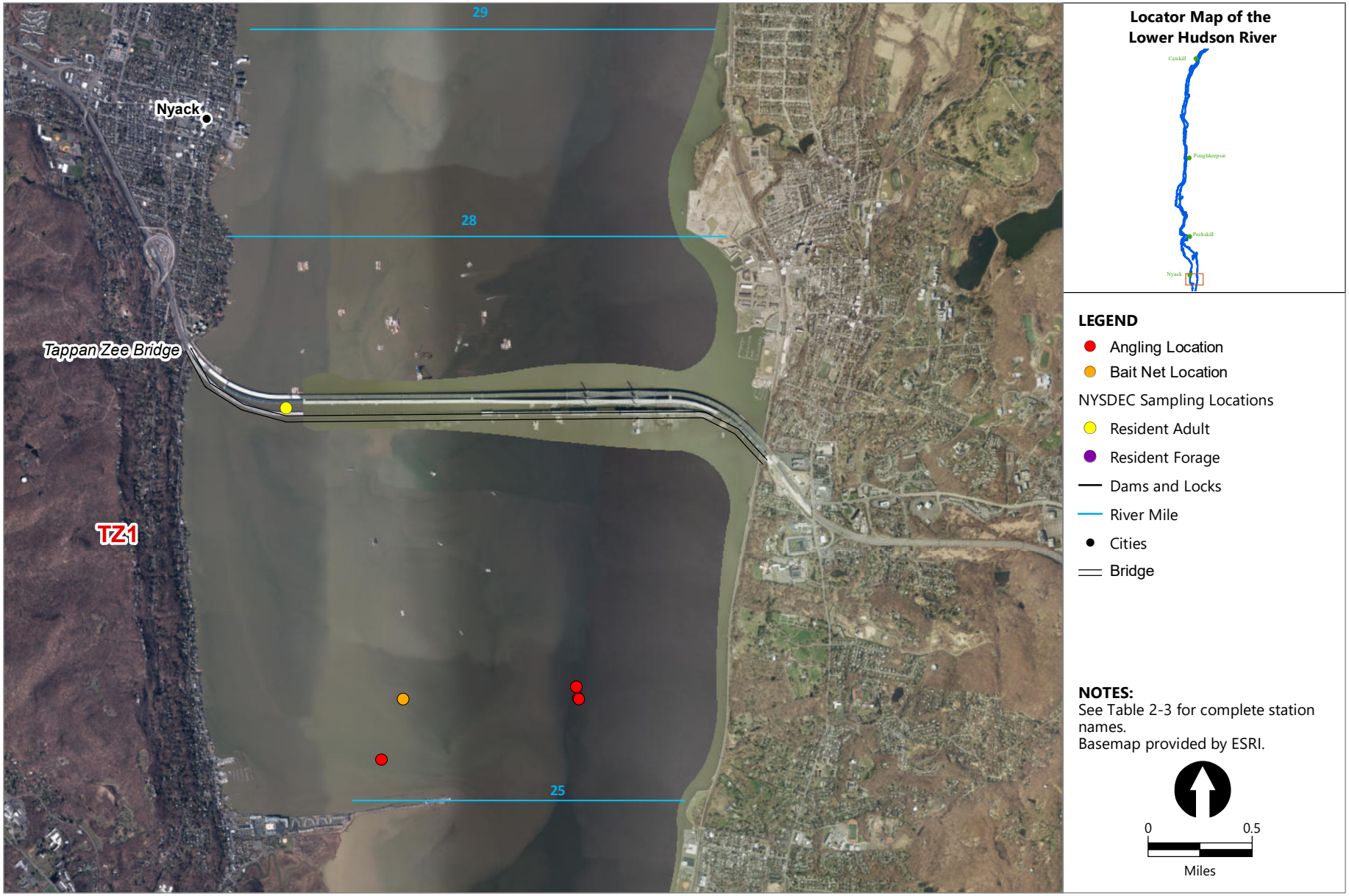

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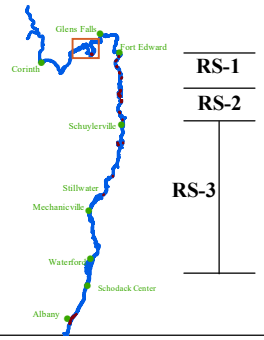
Figure 2-2k
Spring 2017 Fish Sampling Locations
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company

Locator Map of the Upper Hudson River



LEGEND

- Fall 2017 Sampling Transects

NYSDEC Sampling Locations

- Resident Adult
- Resident Forage
- Dams and Locks
- River Mile
- Cities

NOTES:
See Table 2-4 for complete station names.
Basemap provided by ESRI.

0 0.5
Miles

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Figure 2-3a
Fall 2017 Fish Sampling Locations
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company

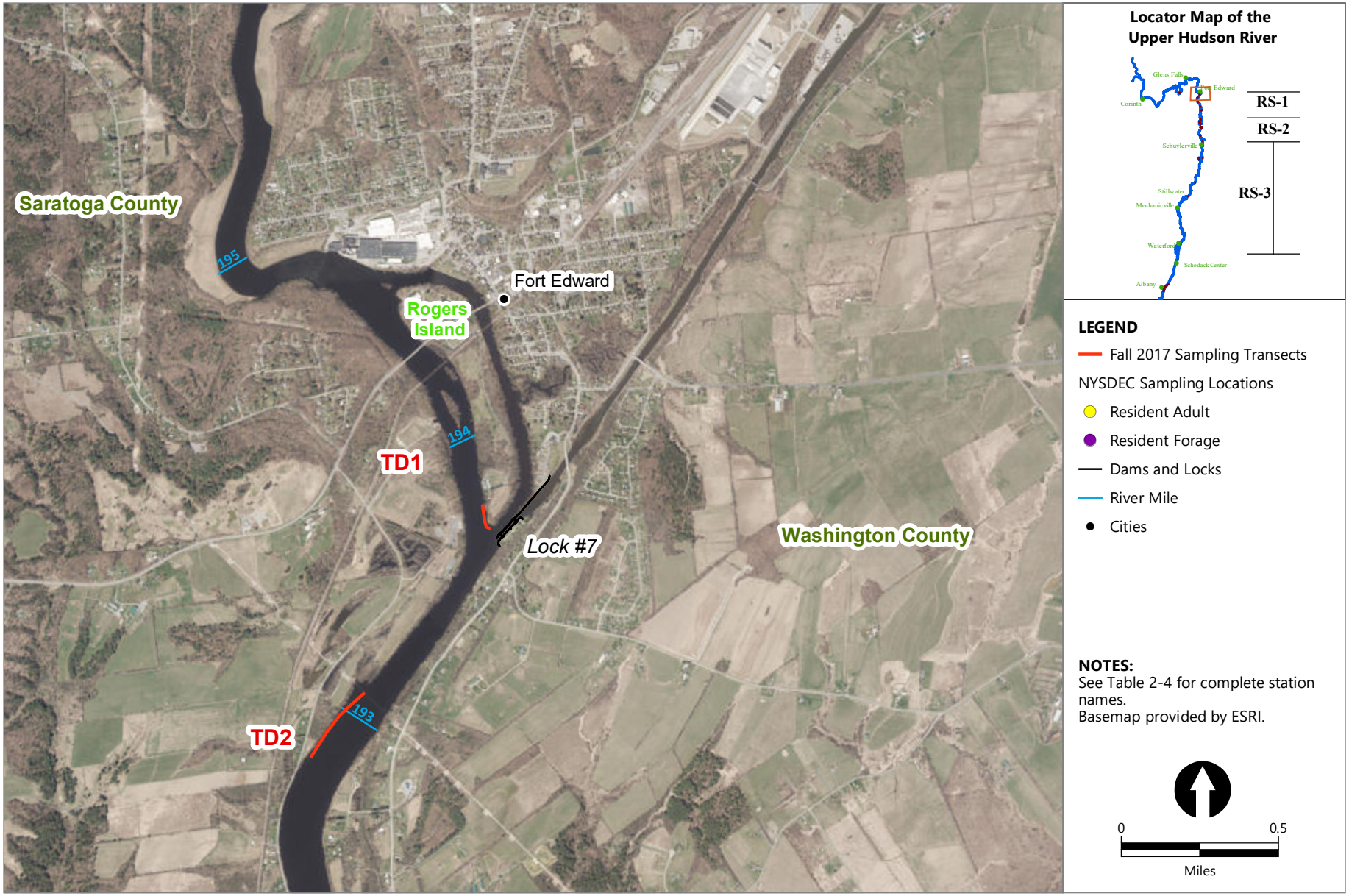

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Figure 2-3b
Fall 2017 Fish Sampling Locations
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company

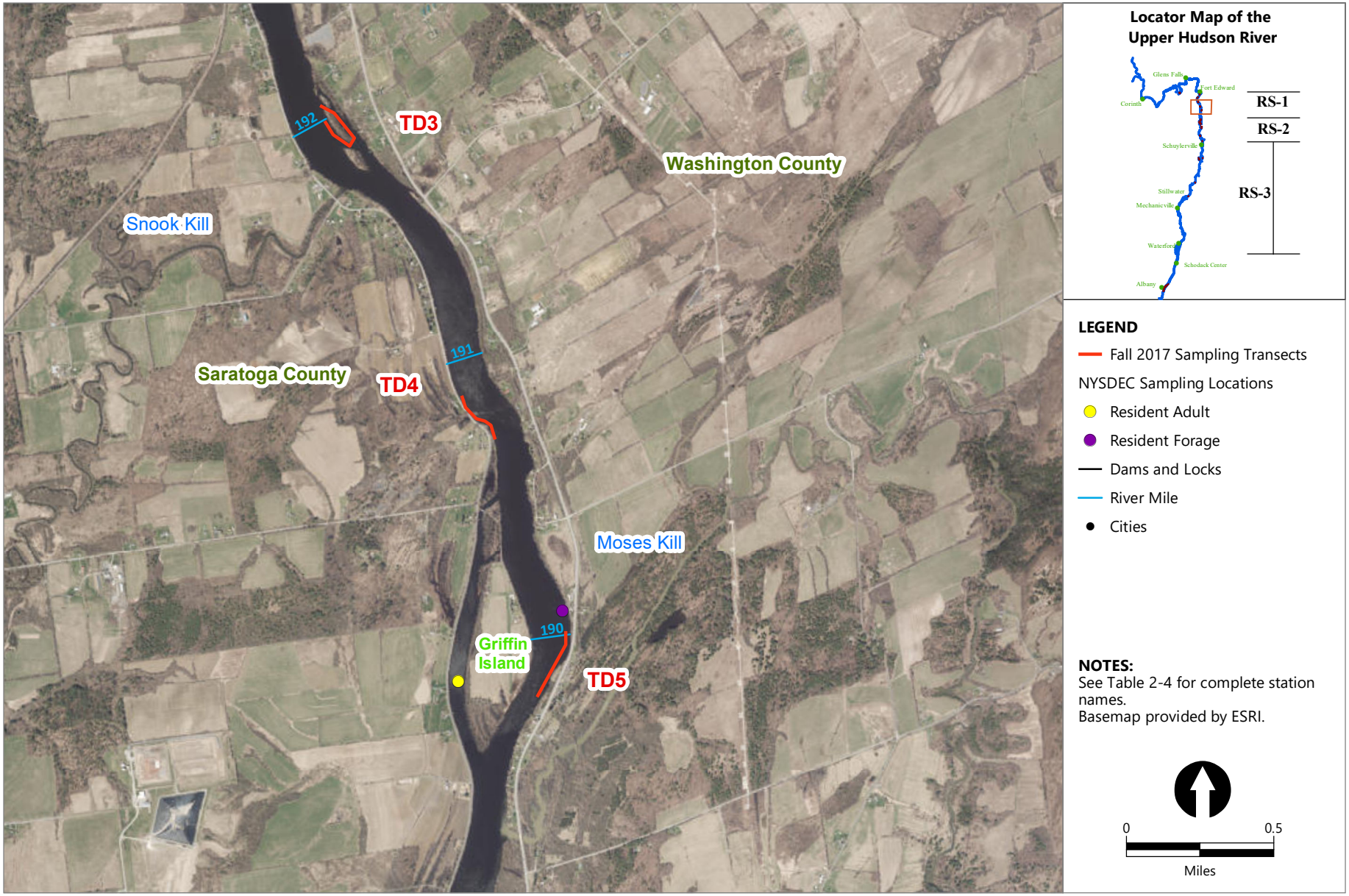

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Figure 2-3c
Fall 2017 Fish Sampling Locations
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company

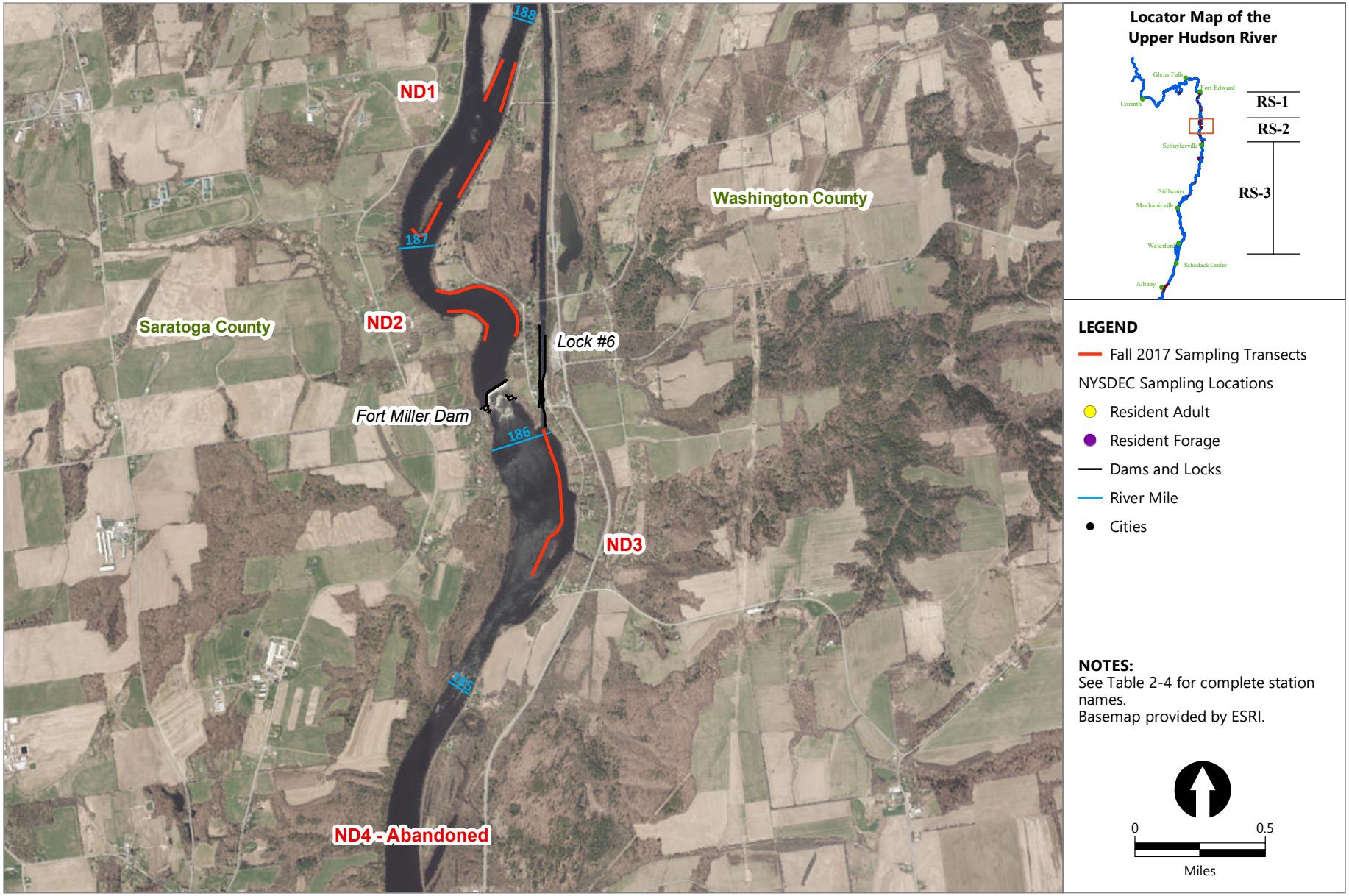

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Figure 2-3d
Fall 2017 Fish Sampling Locations
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company

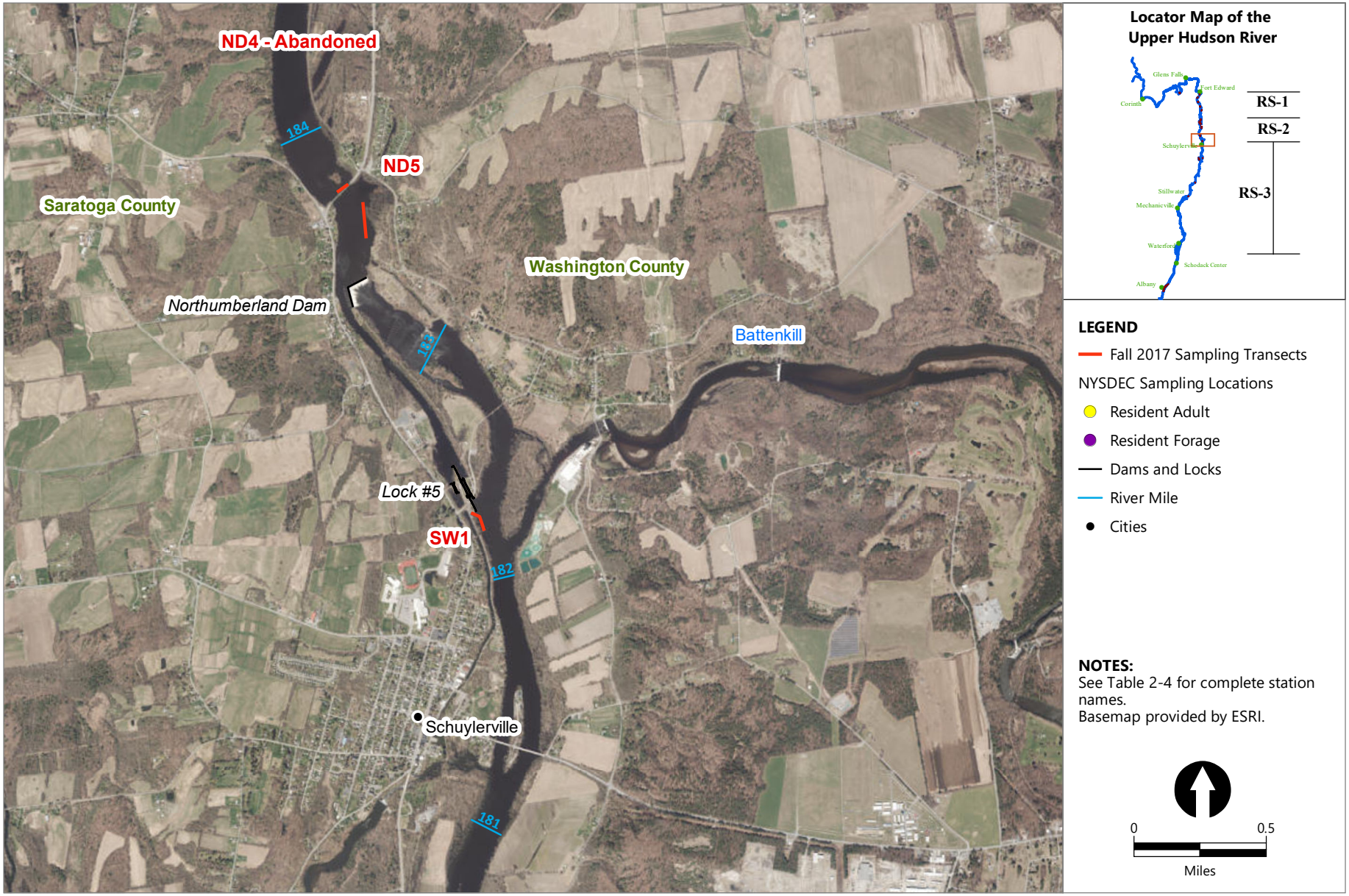

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Figure 2-3e
Fall 2017 Fish Sampling Locations
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company

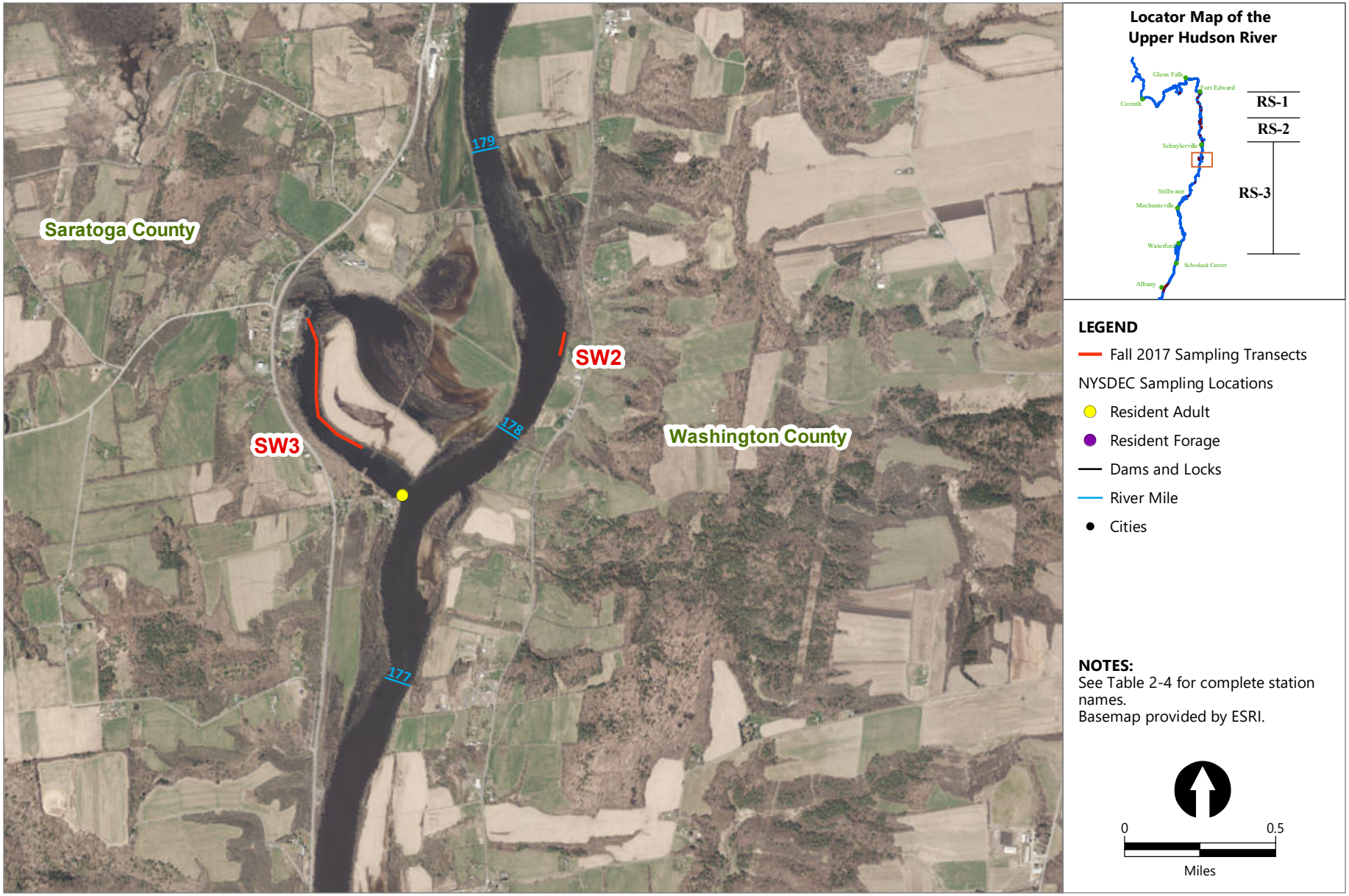

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Figure 2-3f
Fall 2017 Fish Sampling Locations
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company

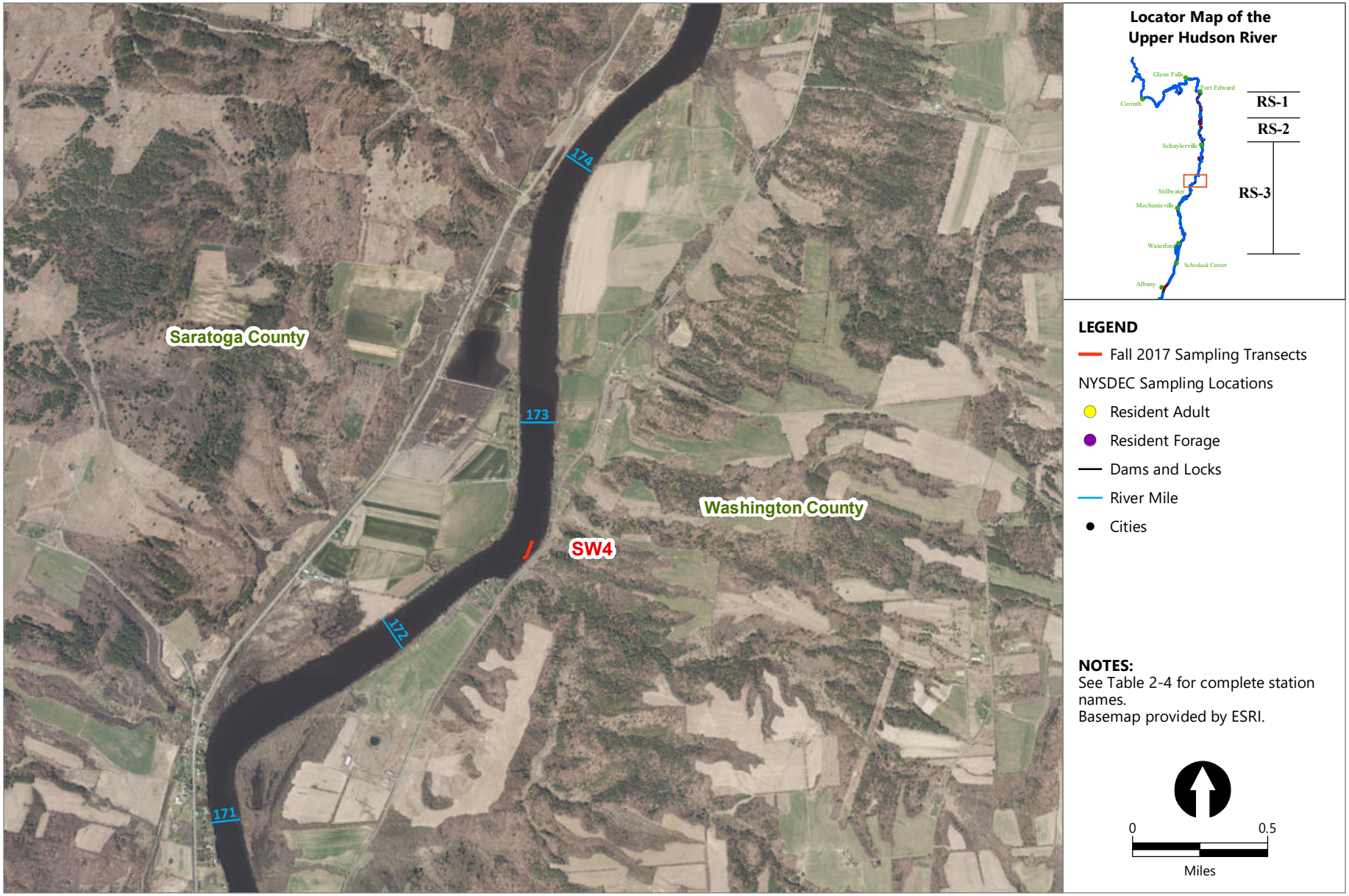

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Figure 2-3g
Fall 2017 Fish Sampling Locations
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company

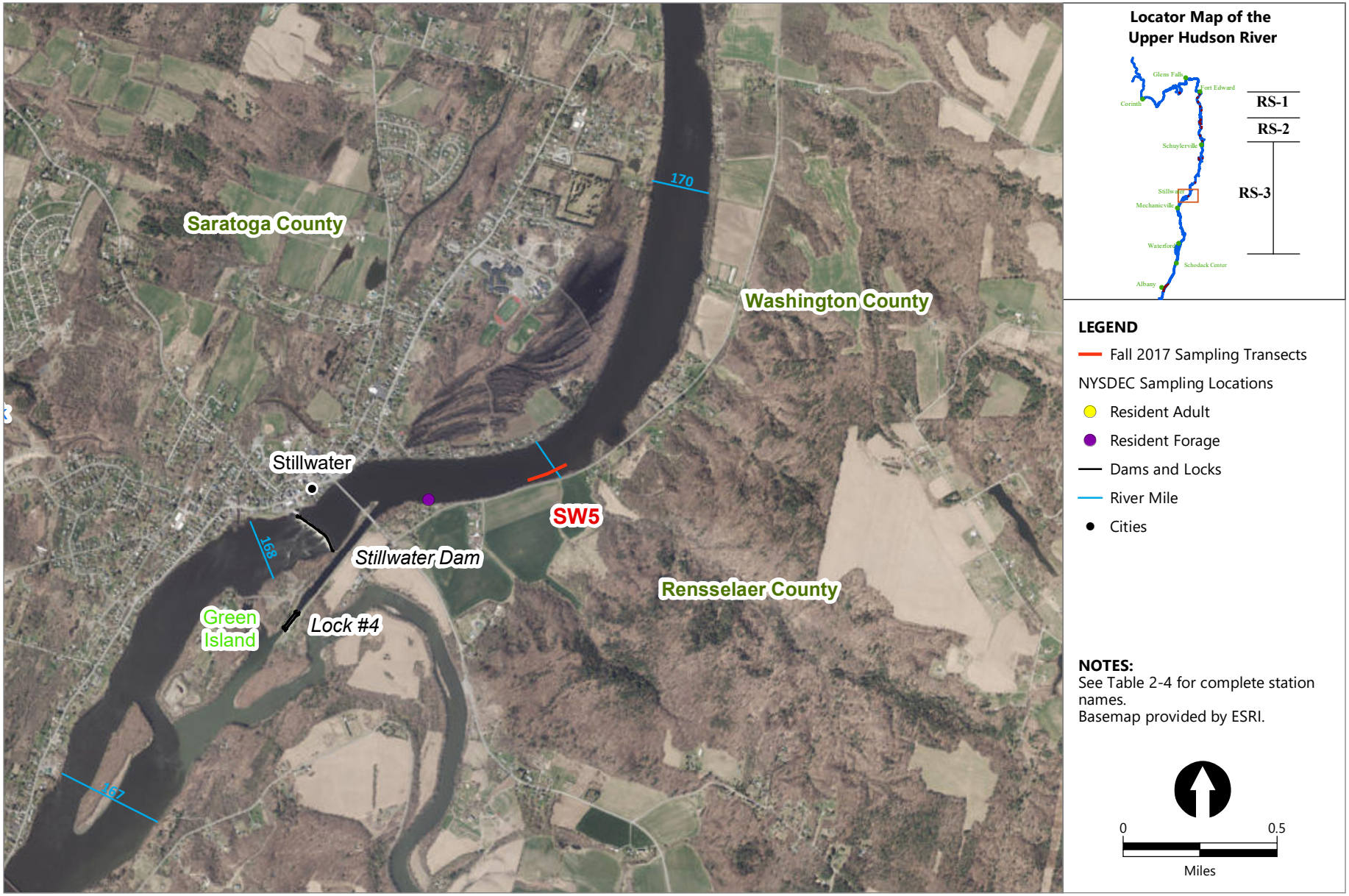

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Figure 2-3h
Fall 2017 Fish Sampling Locations
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company

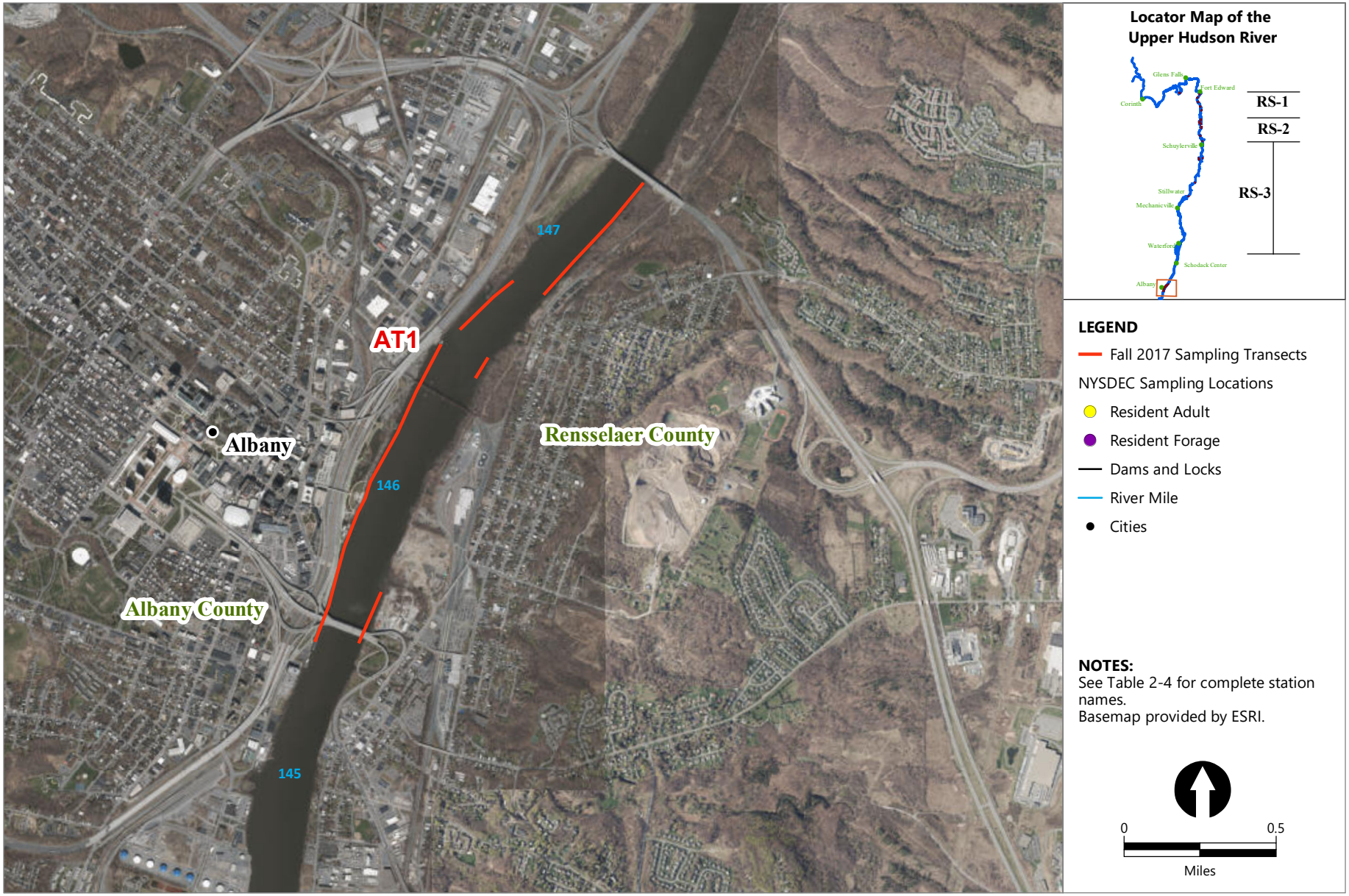

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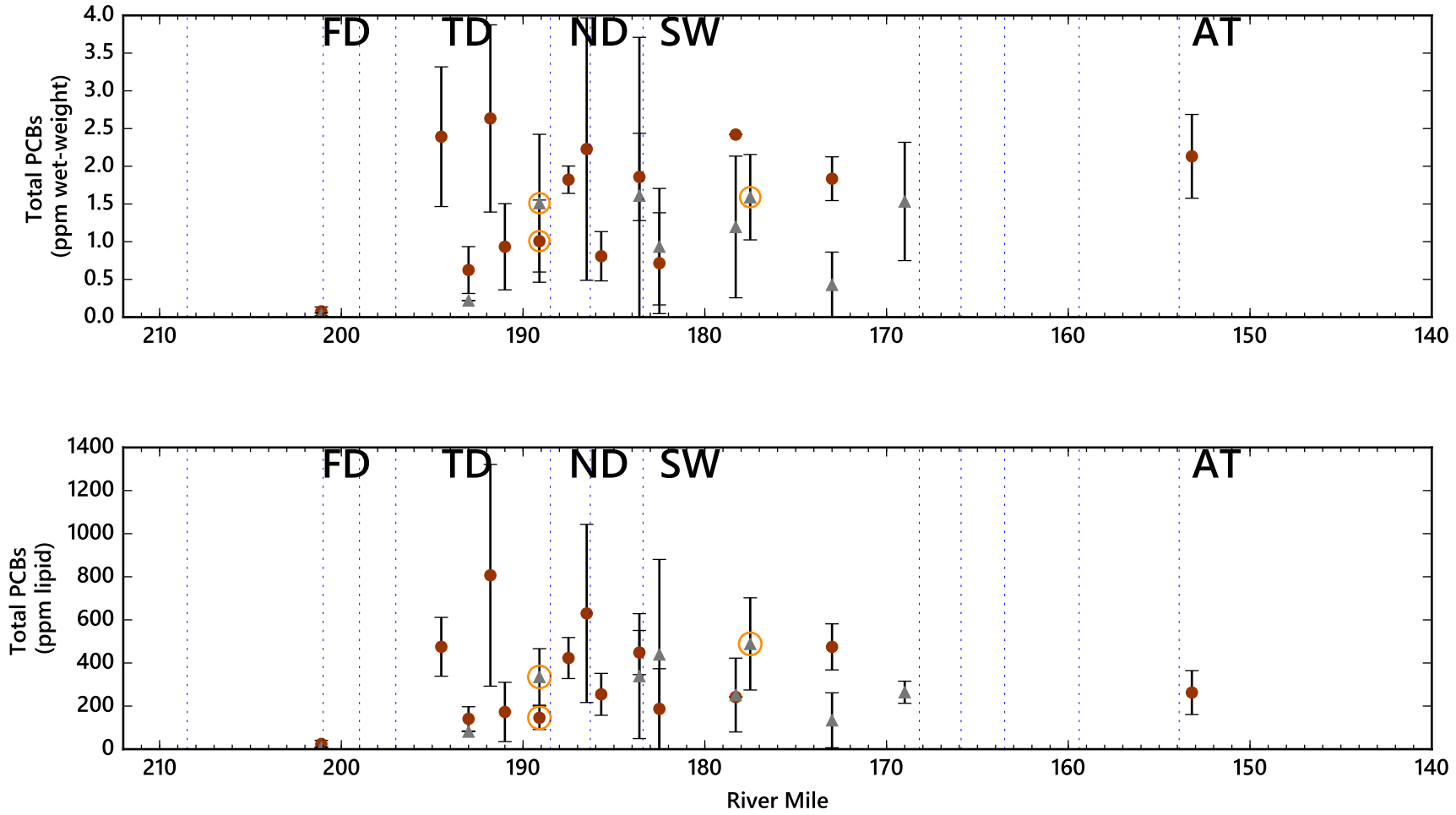
Figure 2-3i
Fall 2017 Fish Sampling Locations
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company

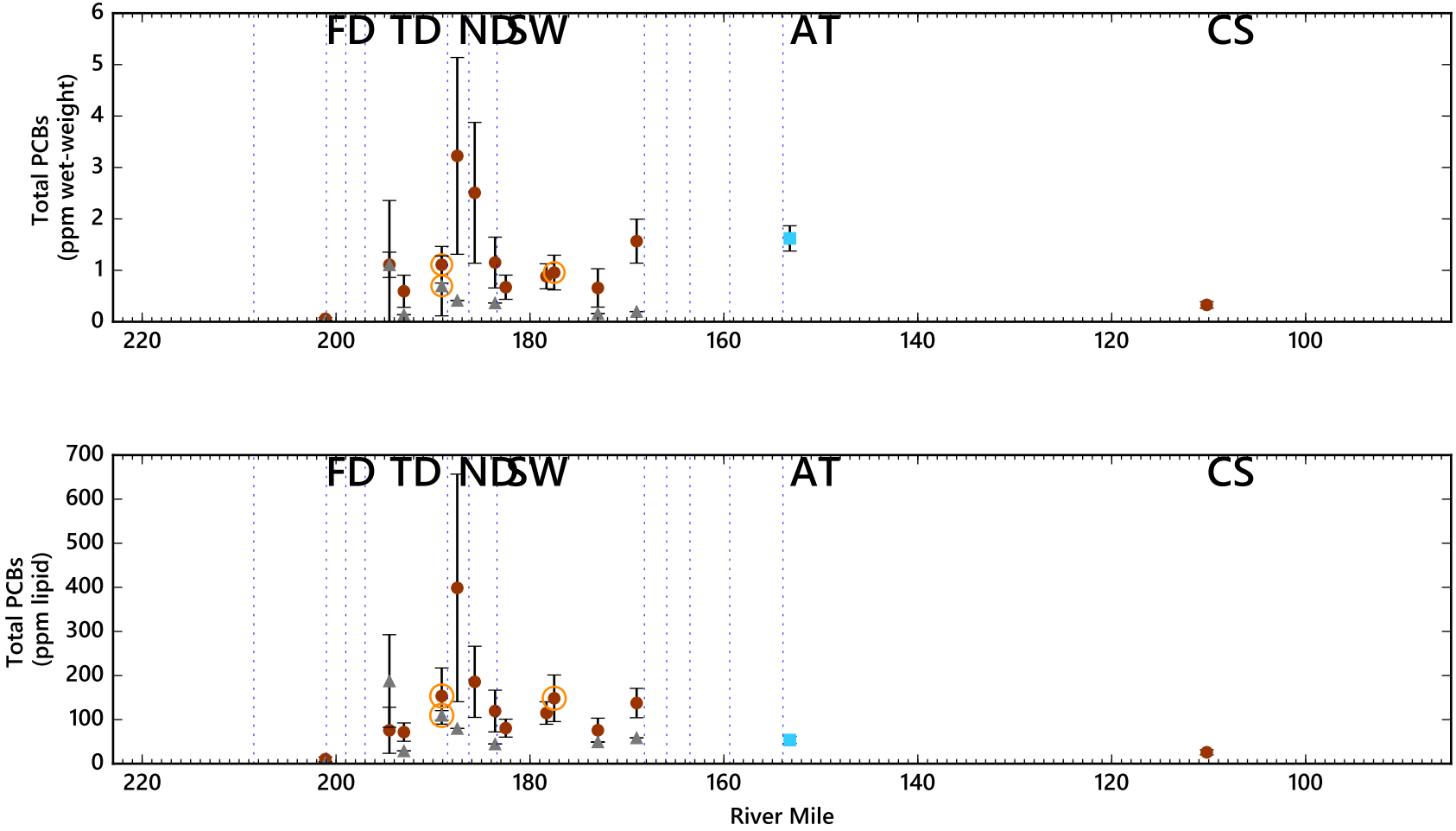


Data points represent arithmetic mean +/- 2 standard error of the mean.
Non-detects set to 1/2 method detection limit.
Year: 2017. Prep: fillet with ribcage.
Blue dotted lines indicate approximate dam locations. Orange circles indicate historical sampling locations.

Publish Date: 08/20/2019 14:16 PM | User: SYR-MKOH
File Path: H:\E_drive\Projects\GE_Hudson\RAMP\Working\RAMP_Fish\Analysis\Python\Spatial\Fish_pcb_Spatial_2019.py

ANCHOR QEA

Figure 4-1
Spatial Patterns in PCB Concentrations in Black Bass
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company



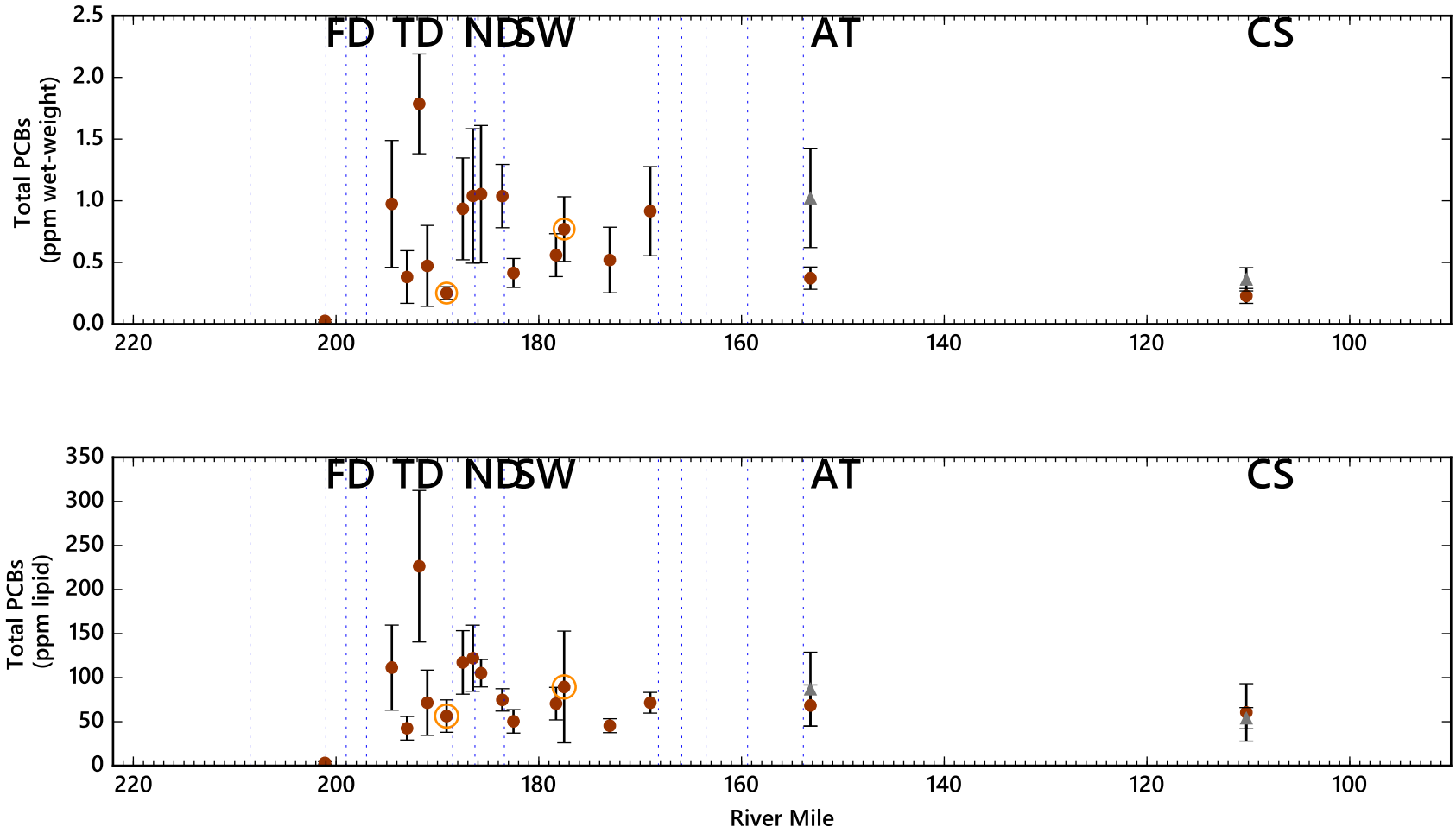
Data points represent arithmetic mean +/- 2 standard error of the mean.
Non-detects set to 1/2 method detection limit.
Year: 2017. Prep: fillet with ribcage.
Blue dotted lines indicate approximate dam locations. Orange circles indicate historical sampling locations.

- Brown Bullhead
- Yellow Bullhead
- Channel Catfish

Publish Date: 08/20/2019 14:16 PM | User: SYR-MKOH
File Path: H:\E_drive\Projects\GE_Hudson\RAMP\Working\RAMP_Fish\Analysis\Python\Spatial\Fish_pcb_Spatial_2019.py

ANCHOR QEA

Figure 4-2
Spatial Patterns in PCB Concentrations in Ictalurids
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company



Data points represent arithmetic mean +/- 2 standard error of the mean.
Non-detects set to 1/2 method detection limit.
Year: 2017. Prep: fillet with ribcage.
Blue dotted lines indicate approximate dam locations. Orange circles indicate historical sampling locations.

Yellow Perch
White Perch

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File Path: H:\E_drive\Projects\GE_Hudson\RAMP\Working\RAMP_Fish\Analysis\Python\Spatial\Fish_pcb_Spatial_2019.py

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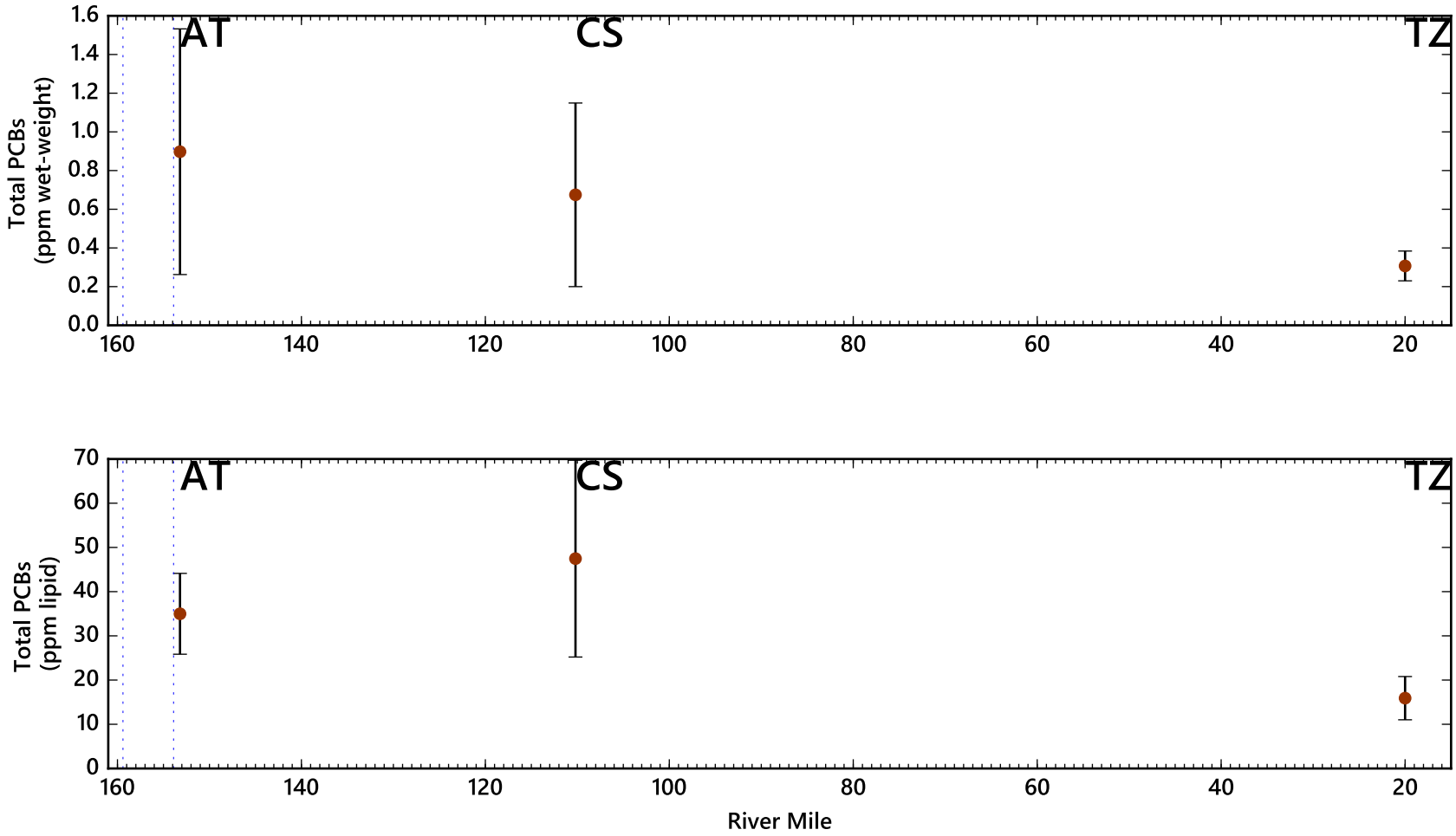
Figure 4-3
Spatial Patterns in PCB Concentrations in Perch
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company

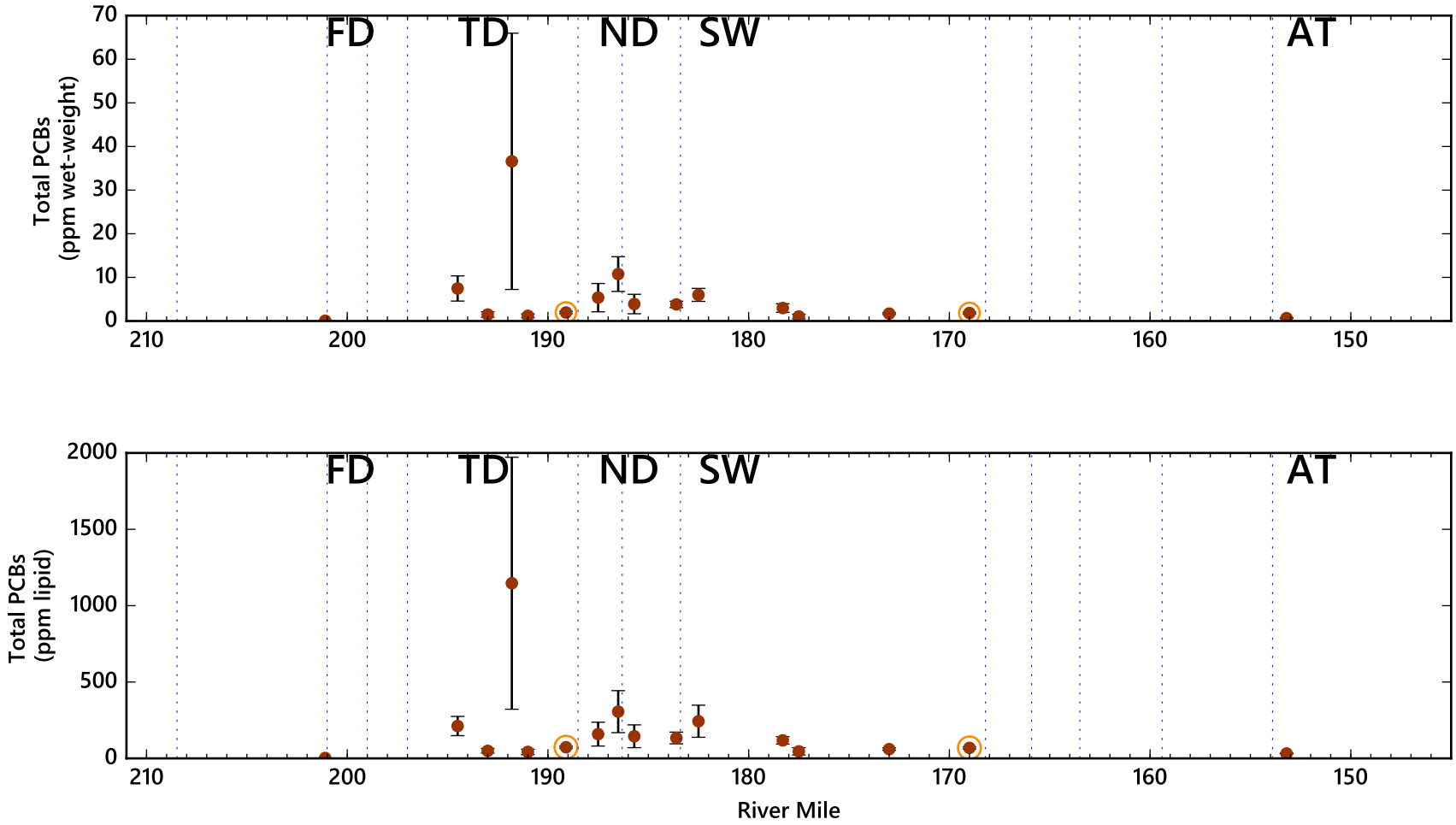


Data points represent arithmetic mean +/- 2 standard error of the mean.
Non-detects set to 1/2 method detection limit.
Year: 2017. Prep: fillet with ribcage.
Blue dotted lines indicate approximate dam locations. Orange circles indicate historical sampling locations.

Publish Date: 08/20/2019 14:16 PM | User: SYR-MKOH
File Path: H:\E_drive\Projects\GE_Hudson\RAMP\Working\RAMP_Fish\Analysis\Python\Spatial\Fish_pcb_Spatial_2019.py

ANCHOR QEA

Figure 4-4
Spatial Patterns in PCB Concentrations in Striped Bass
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company

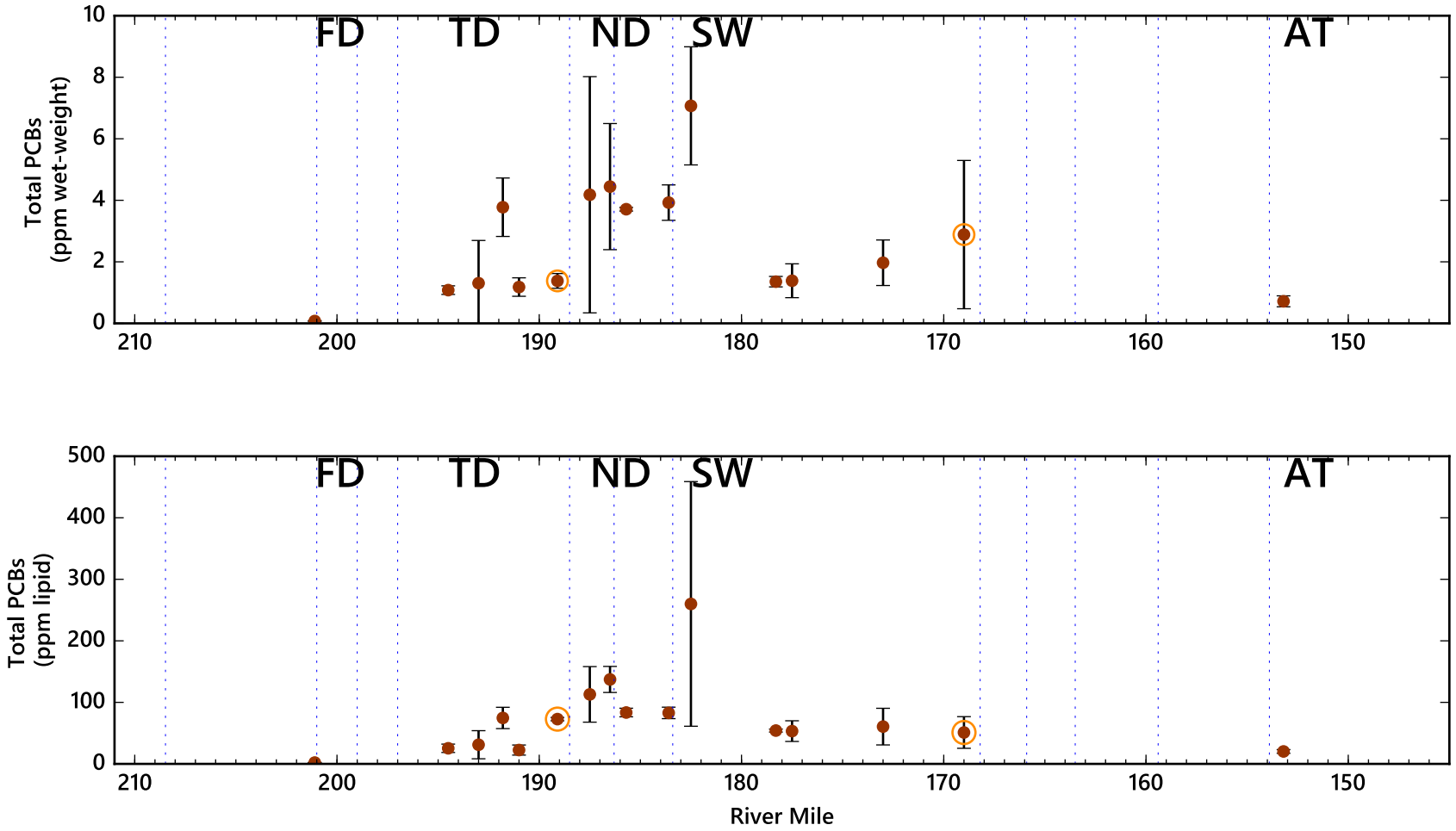


Data points represent arithmetic mean +/- 2 standard error of the mean.
Non-detects set to 1/2 method detection limit.
Year: 2017. Prep: fillet with ribcage.
Blue dotted lines indicate approximate dam locations. Orange circles indicate historical sampling locations.

Publish Date: 08/20/2019 14:16 PM | User: SYR-MKOH
File Path: H:\E_drive\Projects\GE_Hudson\RAMP\Working\RAMP_Fish\Analysis\Python\Spatial\Fish_pcb_Spatial_2019.py

ANCHOR QEA

Figure 4-5
Spatial Patterns in PCB Concentrations in Pumpkinseed
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company

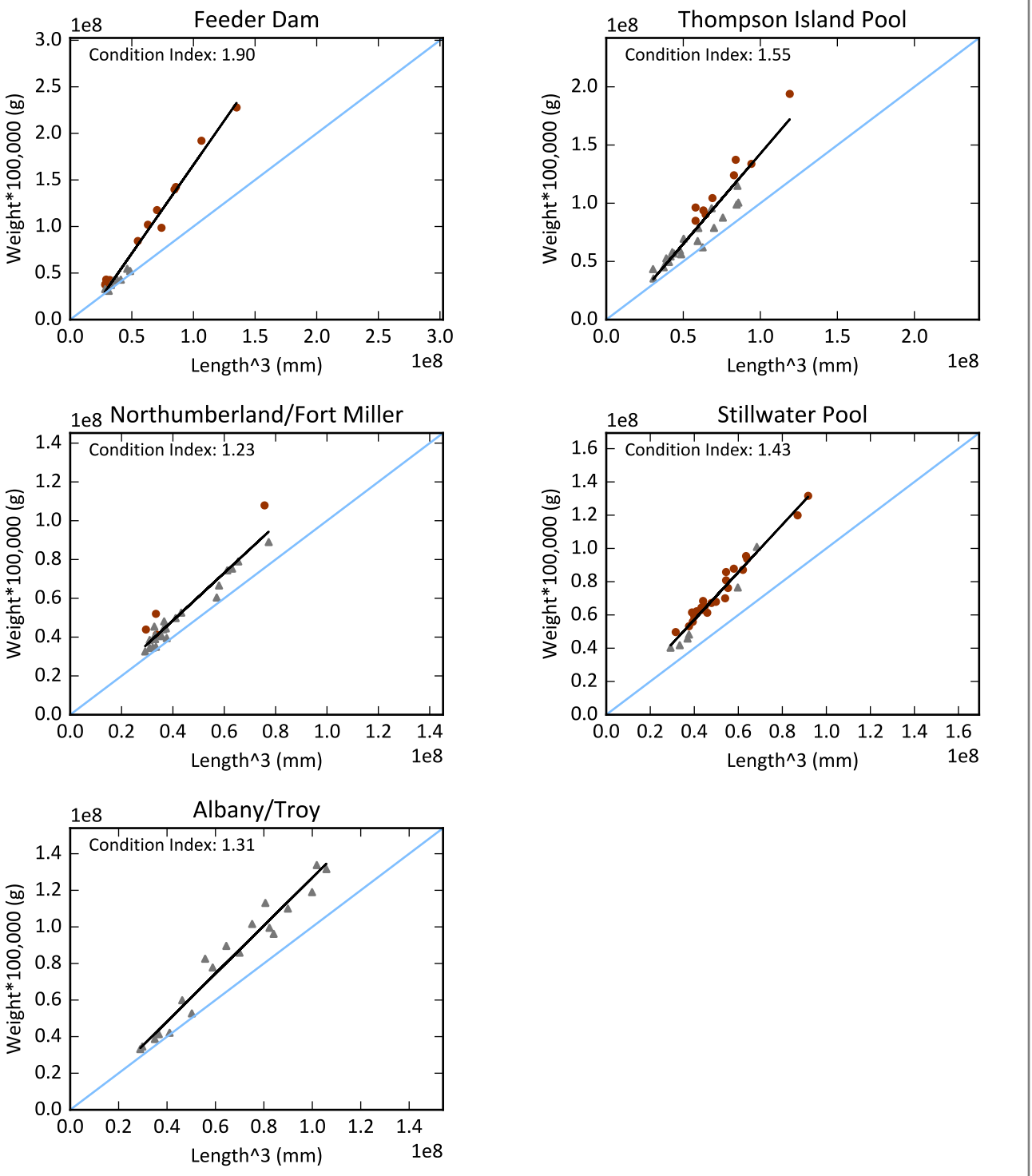


Data points represent arithmetic mean +/- 2 standard error of the mean.
Non-detects set to 1/2 method detection limit.
Year: 2017. Prep: fillet with ribcage.
Blue dotted lines indicate approximate dam locations. Orange circles indicate historical sampling locations.

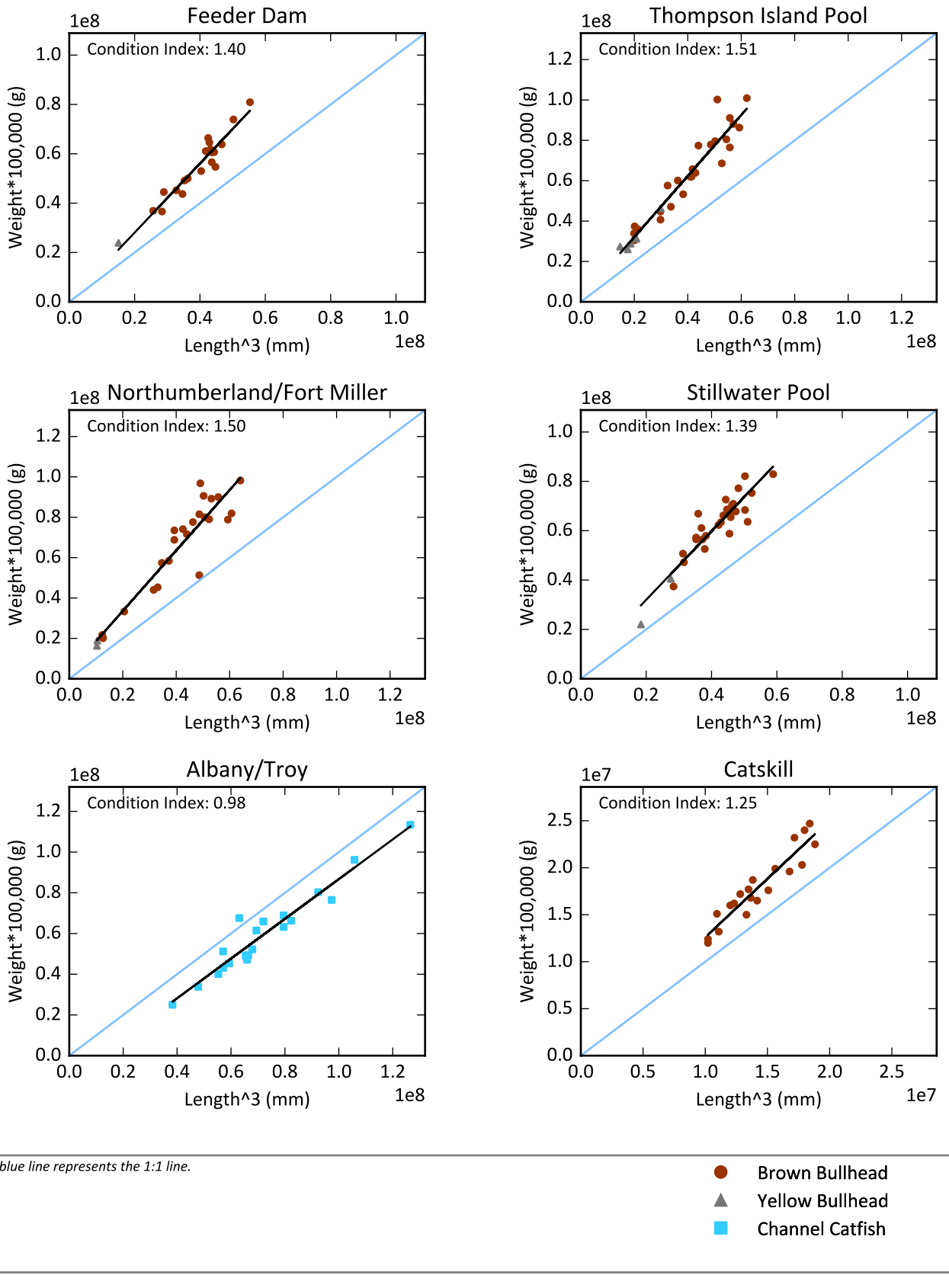
Publish Date: 08/20/2019 14:16 PM | User: SYR-MKOH
File Path: H:\E_drive\Projects\GE_Hudson\RAMP\Working\RAMP_Fish\Analysis\Python\Spatial\Fish_pcb_Spatial_2019.py

ANCHOR QEA

Figure 4-6
Spatial Patterns in PCB Concentrations in Forage Fish
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company

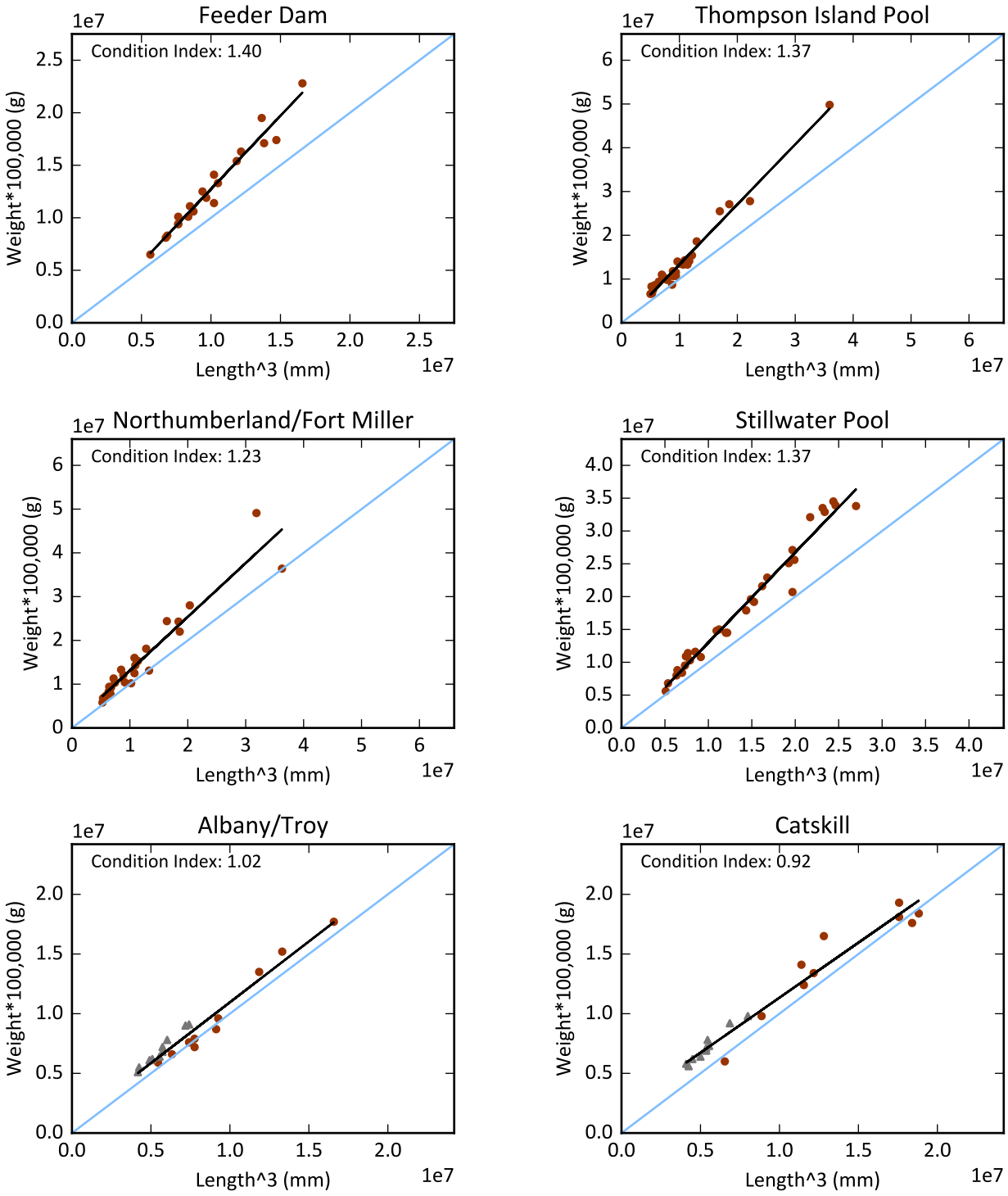


Publish Date: 08/21/2019 11:15 AM | User: SYR-MBEL
File Path: H:\E_drive\Projects\GE_Hudson\RAMP\Working\RAMP_Fish\Analysis\Python\Condition Index\Condition_Index_2019.py



Publish Date: 08/21/2019 11:15 AM | User: SYR-MBEL
File Path: H:\E_drive\Projects\GE_Hudson\RAMP\Working\RAMP_Fish\Analysis\Python\Condition Index\Condition_Index_2019.py

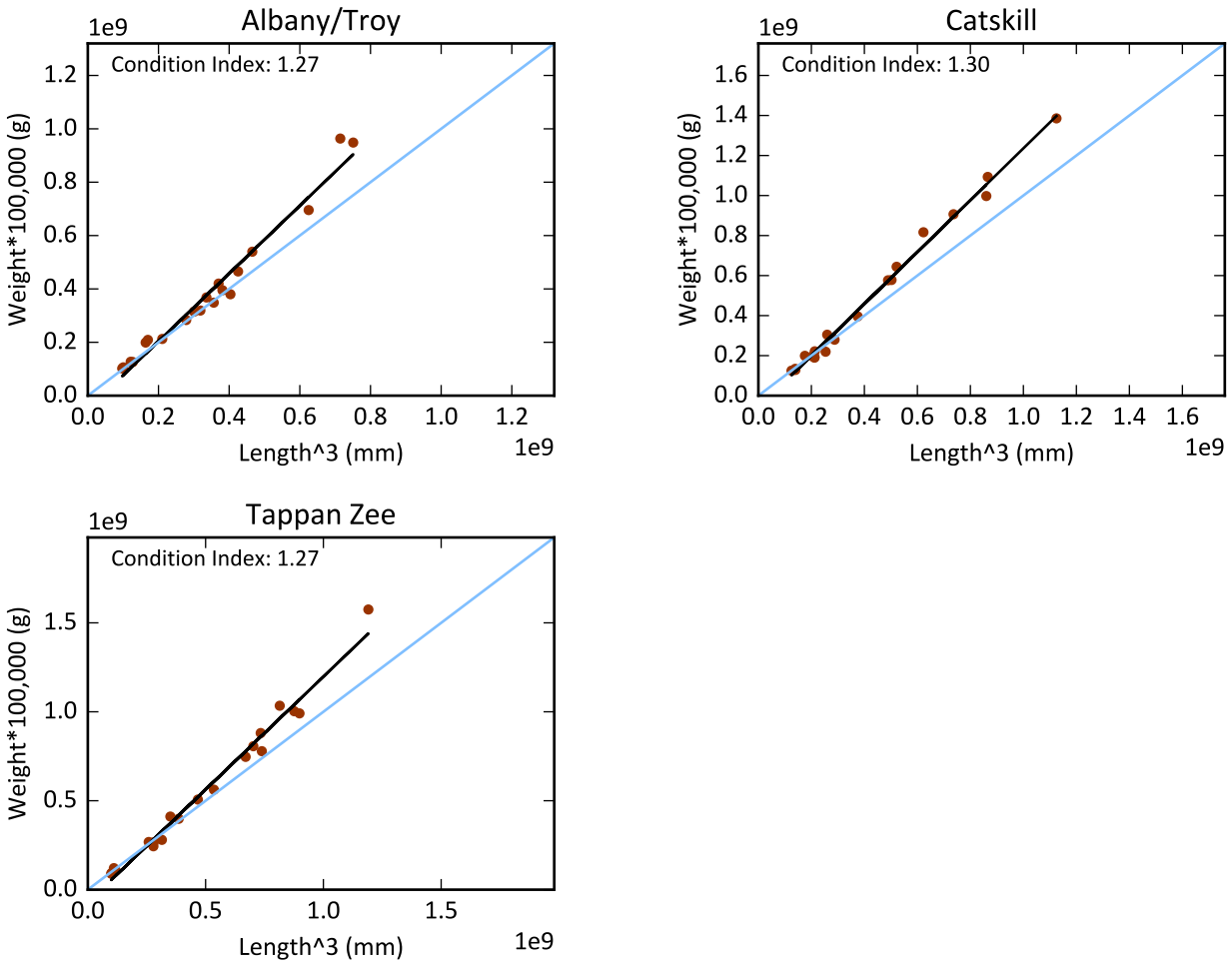
Figure 4-8
Condition Index of Ictalurids for 2017 Sampling Events
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company



Publish Date: 08/21/2019 11:15 AM | User: SYR-MBEL
File Path: H:\E_drive\Projects\GE_Hudson\RAMP\Working\RAMP_Fish\Analysis\Python\Condition Index\Condition_Index_2019.py

ANCHOR QEA

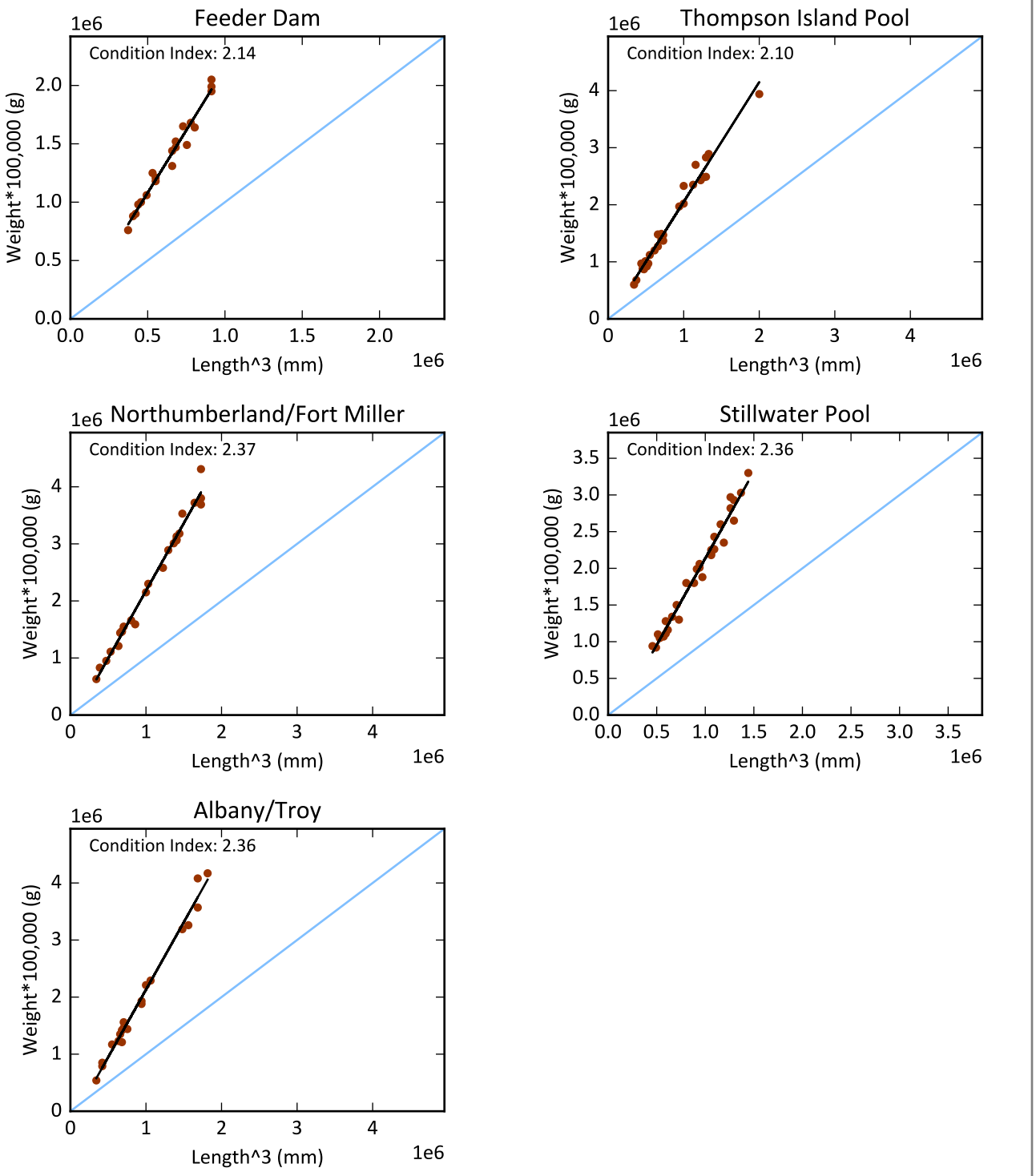
Figure 4-9
Condition Index of Perch for 2017 Sampling Events
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company



Light blue line represents the 1:1 line.

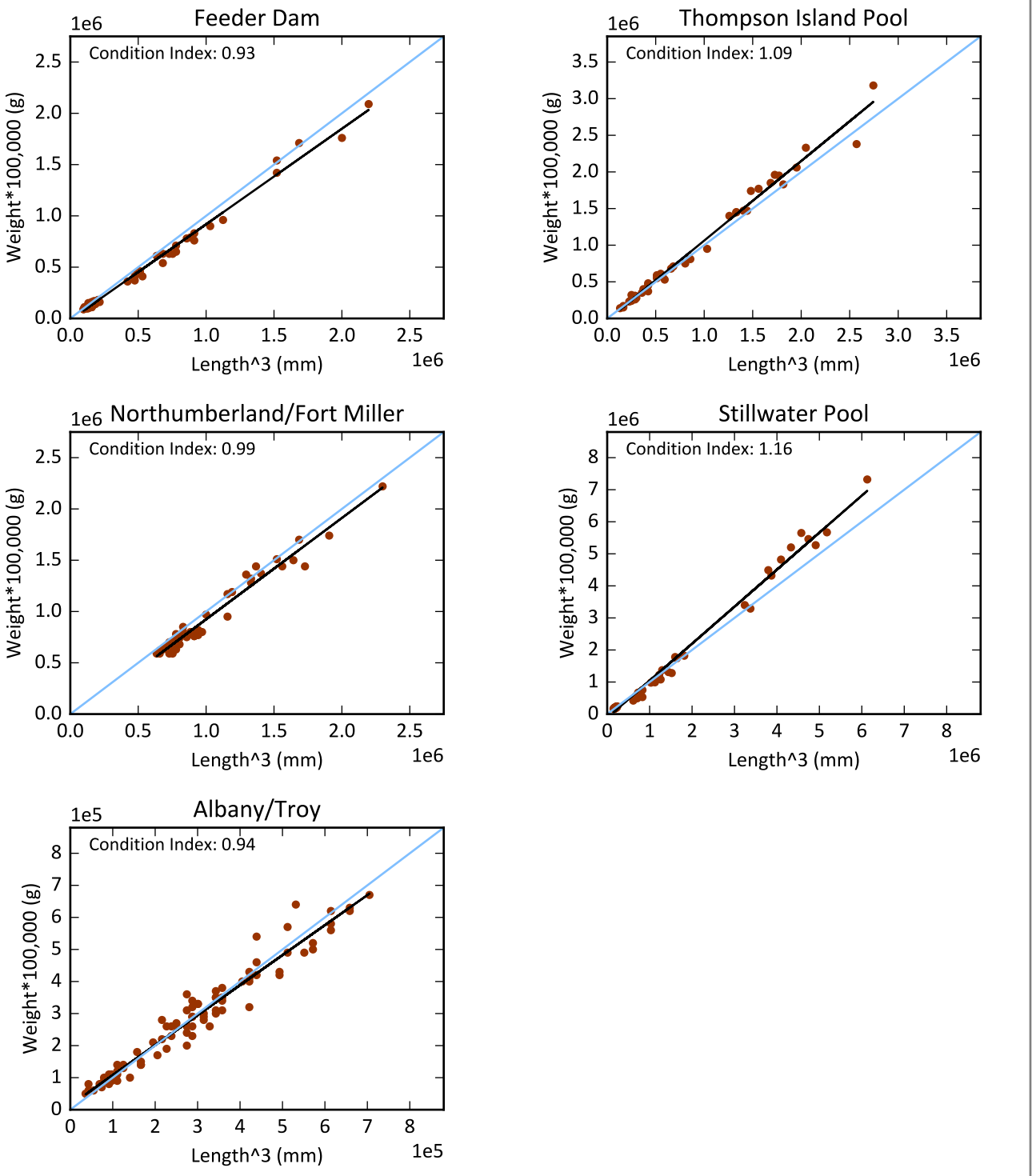
Publish Date: 08/21/2019 11:15 AM | User: SYR-MBEL
File Path: H:\E_drive\Projects\GE_Hudson\RAMP\Working\RAMP_Fish\Analysis\Python\Condition Index\Condition_Index_2019.py

Figure 4-10
Condition Index of Striped Bass for 2017 Sampling Events
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company



Light blue line represents the 1:1 line.

Publish Date: 08/21/2019 11:15 AM | User: SYR-MBEL
File Path: H:\E_drive\Projects\GE_Hudson\RAMP\Working\RAMP_Fish\Analysis\Python\Condition Index\Condition_Index_2019.py



Light blue line represents the 1:1 line.

Publish Date: 08/21/2019 11:15 AM | User: SYR-MBEL
File Path: H:\E_drive\Projects\GE_Hudson\RAMP\Working\RAMP_Fish\Analysis\Python\Condition Index\Condition_Index_2019.py

Figure 4-12
Condition Index of Forage Fish for 2017 Sampling Events
2017 Water and Fish Data Summary Report
Prepared for the General Electric Company

Appendix A
Corrective Action Memoranda and Technical Memoranda

(Provided on accompanying DVD)

From: "Klawinski, Gary J" <Klawinski.Gary@epa.gov>
Date: July 11, 2017 at 8:16:48 AM EDT
To: "Gibson, Bob (GE Corporate)" <bob.gibson@ge.com>
Cc: "Cheplowitz, Michael" <Cheplowitz.Michael@epa.gov>, "King, David" <King.David@epa.gov>, "Kevin Farrar - NYS Department of Environmental Conservation" <kevin.farrar@dec.ny.gov>, Mike Traynor <MTraynor@louisberger.COM>, Bruce Fidler <bfidler@louisberger.com>
Subject: EXT: follow up (approved) RE: Corrective Action Memorandum No. 14 to Phase 2 RAM QAPP

Bob

CAM 14 related to ongoing Hudson River water PCB analysis is acceptable to EPA. Please provide a list of samples being held and a schedule of analysis (with turn times) so that EPA has an understanding of how we will catch up on the backlog.

Thank you,

Gary

From: Gibson, Bob (GE Corporate) [mailto:bob.gibson@ge.com]
Sent: Thursday, May 18, 2017 3:10 PM
To: Klawinski, Gary J <Klawinski.Gary@epa.gov>; Garbarini, Doug <Garbarini.Doug@epa.gov>; Fischer, Douglas <Fischer.Douglas@epa.gov>; 'brian.donohue@usdoj.gov' <brian.donohue@usdoj.gov>; Susan.Edwards@dec.ny.gov; Kevin Farrar <kxfarrar@gw.dec.state.ny.us>; 'alyce.fritz@noaa.gov' (alyce.fritz@noaa.gov) <alyce.fritz@noaa.gov>; Rosman, Lisa (NOAA) <Lisa.Rosman@noaa.gov>; 'bridget.boyd@health.ny.gov' <bridget.boyd@health.ny.gov>; 'Kathryn_Jahn@fws.gov' <Kathryn_Jahn@fws.gov>
Cc: Haggard, John (GE Corporate) <john.haggard@ge.com>; Merrifield, Eric (GE Corporate) <eric.merrifield@ge.com>; Todd, Angelica (GE Corporate) <Angelica.Todd@ge.com>
Subject: RE: Corrective Action Memorandum No. 14 to Phase 2 RAM QAPP

Gary –

I received a few undeliverable emails on yesterday's transmittal of CAM 14; the file may have been too large for some email systems. If you did not receive the CAM, please go to the link on the attached

email from ESI and download a copy. Feel free to give me a call if you have any problems with the download. Thanks.

Bob

From: Gibson, Bob (GE Corporate)
Sent: Wednesday, May 17, 2017 5:26 PM
To: 'klawinski.gary@epa.gov' <klawinski.gary@epa.gov>; 'Garbarini.Doug@epa.gov' <Garbarini.Doug@epa.gov>; Fischer.Douglas@epamail.epa.gov; 'brian.donohue@usdoj.gov' <brian.donohue@usdoj.gov>; 'Susan.Edwards@dec.ny.gov' <Susan.Edwards@dec.ny.gov>; Kevin Farrar <kxfarrar@gw.dec.state.ny.us>; 'alyce.fritz@noaa.gov' (alyce.fritz@noaa.gov) <alyce.fritz@noaa.gov>; 'Lisa.Rosman@noaa.gov' <Lisa.Rosman@noaa.gov>; 'bridget.boyd@health.ny.gov' <bridget.boyd@health.ny.gov>; 'Kathryn_Jahn@fws.gov' <Kathryn_Jahn@fws.gov>
Cc: Haggard, John (GE Corporate) <john.haggard@ge.com>; 'Merrifield, Eric (GE Corporate)' <eric.merrifield@ge.com>; Todd, Angelica (GE Corporate) <Angelica.Todd@ge.com>
Subject: Corrective Action Memorandum No. 14 to Phase 2 RAM QAPP

Gary –

Attached is Corrective Action Memorandum No. 14 to the Phase 2 RAM QAPP. Please let me know if you have any questions or would like to discuss. Thanks.

Bob Gibson
Senior Project Manager - Environmental Remediation
Global Operations, Environment, Health & Safety
GE

T +1 518 388 7505
M +1 518 527 3418
bob.gibson@ge.com

1 River Road – Bldg. 5-7W
Schenectady, NY 12345 United States

GENERAL ELECTRIC COMPANY
HUDSON RIVER PHASE 2 REMEDIAL ACTION MONITORING PROGRAM
2017 CORRECTIVE ACTION MEMORANDUM No. 14

Date: May 17, 2017
Organization Name: Environmental Standards, Inc.
Initiator's Name and Title: Meg Michell

Problem Description: According to Section 2.5.3.3 of the Phase 2 Remedial Action Monitoring Quality Assurance Project Plan (Phase 2 RAM QAPP), water samples collected during the off-season monitoring program were be analyzed for low-level modified Green Bay Method (mGBM) PCBs at Bakers Falls and Rogers Island stations, Aroclor PCBs at the remaining stations, and TSS at all locations in accordance with the procedures and analytical methods specified in Attachment A. According to Section 2 of Attachment A, these analyses were to be performed according to the Pace Analytical, Inc. Schenectady NY (Pace Schenectady) standard operating procedures (SOPs) contained in Appendices A2-1 through A2-10. The Pace Schenectady laboratory facility closed operations at the end of 2016. In addition, the mGBM is not offered at any other commercial laboratory facility. The purpose of this Corrective Action Memorandum (CAM) is to document the analytical procedures that will be used for water samples collected starting at the beginning of 2017 through the beginning of the long-term operation, maintenance, and monitoring (OM&M) program for water, fish and sediment.

Reported To: Bob Gibson, GE

Corrective Action: Pace Analytical Services, Inc. of Minneapolis, Minnesota will perform the analysis for total suspended solids (TSS) following Standard Method 2540D (modified to be consistent with ASTM Method D3977-97 as described in Section 2.4 of Attachment A of the Phase 2 RAM QAPP). This laboratory holds the required New York certification for this analysis as shown in Attachment 1. The SOP used for by the laboratory for TSS analysis is provided as Attachment 2. The laboratory reporting limit (RL) and measurement performance criteria for precision, accuracy/bias, representativeness, comparability, completeness, and sensitivity will be the same as used by Pace Schenectady and summarized in Attachment A Tables A1-2 and A2-1c.

Vista Analytical Laboratory of El Dorado Hills, California will perform the analysis of water samples from all stations for PCBs following US EPA Method 1668C. This laboratory holds the required New York certification for this analysis as shown in Attachment 3. The SOP used by the laboratory for PCB analysis by EPA Method 1668C is provided as Attachment 4. The laboratory method detection limits (MDLs) and RLs are provided in Attachment 5. The measurement performance criteria for precision, accuracy/bias, representativeness, comparability, completeness, and sensitivity are presented in Table 1 to this corrective action memorandum.

The laboratory MDLs have been provided in Attachment 5; however, results will be reported to the sample specific estimated detection limits (EDLs) as calculated in Section 16.2 of the laboratory SOP included Attachment 4. Total PCBs will be calculated as the sum of positive

results that are ≥ the EDL (unless qualified due to blank contamination) with consideration of the following rules:

- Results that are estimated ("J") will be included in the sum
- Not-detected values (EDL "U") will be set to "0"
- Results that do not meet all of the qualitative criteria of EPA Method 1668C and have been qualified as an Estimated Maximum Possible Concentration (EMPC) will be set to "0"

Reviewed and Implemented By: David Blye (Environmental Standards)

Copy to: GE Program Manager: Bob Gibson
QA Program Manager: David Blye (Environmental Standards)

Table 1
Measurement Performance Criteria for Water Samples

Data Quality Indicators (DQIs)[1]	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
Accuracy	< RL, or associated samples >5× blank values	Laboratory or Equipment Blank	S&A
	See Table 7 of Attachment 4	Ongoing Precision and Recovery (OPR)	A
	See Table 7 of Attachment 4	Internal Standards	A
Precision	The RPD for water field duplicates should be ≤35% for results >5x the RL. The difference between results should be ≤ the RL when at least one result is ≤5x the RL	Field Duplicates	S&A
Sensitivity	See Attachment 5 to CAM	Reporting Limits	A
Representativeness	Use of standardized collection and analytical methods	Field audits and laboratory audits. See Phase 2 RAM QAPP Section 10.3.4	S&A
Completeness	95%	See Phase 2 RAM QAPP Section 10.3.6	S&A
Comparability	Based on accuracy and media comparison	Use of standardized SOPs by field and analytical contractors	S&A

Notes

Matrix: Water
Analytical Parameter: Total PCBs as Congeners
Concentration Level: Low to High
Method: EPA 1668C (Attachment 4; Vista SOP 31, Rev. 15)

RL = Reporting Limit; R = Recovery; RPD = Relative Percent Difference

[1]Data Quality Indicators (a.k.a. PASRCC parameters, i.e., precision, accuracy/bias, sensitivity, representativeness, data completeness, comparability).

ATTACHMENT 1

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2018
Issued April 01, 2017

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MS. SARAH CHERNEY
PACE ANALYTICAL SERVICES, LLC - MINNEAPOLIS MN
1700 ELM STREET SE SUITE 200
MINNEAPOLIS, MN 55414-2485

NY Lab Id No: 11647

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES POTABLE WATER
All approved analytes are listed below:

Metals I	
Arsenic, Total	EPA 200.8 Rev. 5.4
Barium, Total	EPA 200.8 Rev. 5.4
Cadmium, Total	EPA 200.8 Rev. 5.4
Chromium, Total	EPA 200.8 Rev. 5.4
Copper, Total	EPA 200.8 Rev. 5.4
Lead, Total	EPA 200.8 Rev. 5.4
Manganese, Total	EPA 200.8 Rev. 5.4
Mercury, Total	EPA 245.1 Rev. 3.0
	EPA 200.8 Rev. 5.4
Selenium, Total	EPA 200.8 Rev. 5.4
Silver, Total	EPA 200.8 Rev. 5.4
Zinc, Total	EPA 200.8 Rev. 5.4
Metals II	
Aluminum, Total	EPA 200.8 Rev. 5.4
Antimony, Total	EPA 200.8 Rev. 5.4
Beryllium, Total	EPA 200.8 Rev. 5.4
Nickel, Total	EPA 200.8 Rev. 5.4
Thallium, Total	EPA 200.8 Rev. 5.4
Metals III	
Uranium (Mass)	EPA 200.8 Rev. 5.4
Miscellaneous	
2,3,7,8-Tetrachlorodibenzo-p-dioxin	EPA 1613B

Serial No.: 56113

Property of the New York State Department of Health. Certificates are valid only at the address shown, must be conspicuously posted, and are printed on secure paper. Continued accreditation depends on successful ongoing participation in the Program. Consumers are urged to call (518) 485-5570 to verify the laboratory's accreditation status.

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WADSWORTH CENTER

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NY Lab Id No: 11647

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All approved analytes are listed below:

Dioxins and Furans	
1,2,3,4,6,7,8,9-Octachlorodibenzofuran	EPA 8290A
	EPA 1613B
1,2,3,4,6,7,8,9-Octachlorodibenzo-p-diox	EPA 8290A
	EPA 1613B
1,2,3,4,6,7,8-Heptachlorodibenzofuran	EPA 8290A
	EPA 1613B
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxi	EPA 8290A
	EPA 1613B
1,2,3,4,7,8,9-Heptachlorodibenzofuran	EPA 8290A
	EPA 1613B
1,2,3,4,7,8-Hexachlorodibenzofuran	EPA 8290A
	EPA 1613B
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	EPA 8290A
	EPA 1613B
1,2,3,6,7,8-Hexachlorodibenzofuran	EPA 8290A
	EPA 1613B
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	EPA 8290A
	EPA 1613B
1,2,3,7,8,9-Hexachlorodibenzofuran	EPA 8290A
	EPA 1613B
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	EPA 8290A
	EPA 1613B
1,2,3,7,8-Pentachlorodibenzofuran	EPA 8290A
	EPA 1613B
1,2,3,7,8-Pentachlorodibenzo-p-dioxin	EPA 8290A
	EPA 1613B

Dioxins and Furans	
2,3,4,6,7,8-Hexachlorodibenzofuran	EPA 8290A
	EPA 1613B
2,3,4,7,8-Pentachlorodibenzofuran	EPA 8290A
	EPA 1613B
2,3,7,8-Tetrachlorodibenzofuran	EPA 8290A
	EPA 1613B
2,3,7,8-Tetrachlorodibenzo-p-dioxin	EPA 8290A
	EPA 1613B
Metals I	
Barium, Total	EPA 200.7 Rev. 4.4
	EPA 6010C
	EPA 6020A
	EPA 200.8 Rev. 5.4
Cadmium, Total	EPA 200.7 Rev. 4.4
	EPA 6010C
	EPA 6020A
	EPA 200.8 Rev. 5.4
Calcium, Total	EPA 200.7 Rev. 4.4
	EPA 6010C
	EPA 6020A
	EPA 200.8 Rev. 5.4
Chromium, Total	EPA 200.7 Rev. 4.4
	EPA 6010C
	EPA 6020A
	EPA 200.8 Rev. 5.4

Serial No.: 56114

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All approved analytes are listed below:

Metals I		**Metals I**	
Copper, Total	EPA 200.7 Rev. 4.4	Potassium, Total	EPA 6010C
	EPA 6010C		EPA 6020A
	EPA 6020A		EPA 200.8 Rev. 5.4
	EPA 200.8 Rev. 5.4	Silver, Total	EPA 200.7 Rev. 4.4
Iron, Total	SM 3500-Fe B-97,-11		EPA 6010C
	EPA 200.7 Rev. 4.4		EPA 6020A
	EPA 6010C		EPA 200.8 Rev. 5.4
	EPA 6020A	Sodium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4		EPA 6010C
Lead, Total	EPA 200.7 Rev. 4.4		EPA 6020A
	EPA 6010C		EPA 200.8 Rev. 5.4
	EPA 6020A	Strontium, Total	EPA 6020A
	EPA 200.8 Rev. 5.4		EPA 200.8 Rev. 5.4
Magnesium, Total	EPA 200.7 Rev. 4.4	**Metals II**	
	EPA 6010C	Aluminum, Total	EPA 200.7 Rev. 4.4
	EPA 6020A		EPA 6010C
	EPA 200.8 Rev. 5.4		EPA 6020A
Manganese, Total	EPA 200.7 Rev. 4.4		EPA 200.8 Rev. 5.4
	EPA 6010C	Antimony, Total	EPA 200.7 Rev. 4.4
	EPA 6020A		EPA 6010C
	EPA 200.8 Rev. 5.4		EPA 6020A
Nickel, Total	EPA 200.7 Rev. 4.4		EPA 200.8 Rev. 5.4
	EPA 6010C	Arsenic, Total	EPA 200.7 Rev. 4.4
	EPA 6020A		EPA 6010C
	EPA 200.8 Rev. 5.4		EPA 6020A
Potassium, Total	EPA 200.7 Rev. 4.4		

Serial No.: 56114

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All approved analytes are listed below:

Metals II

Arsenic, Total	EPA 200.8 Rev. 5.4
Beryllium, Total	EPA 200.7 Rev. 4.4
	EPA 6010C
	EPA 6020A
	EPA 200.8 Rev. 5.4
Mercury, Total	EPA 7470A
Selenium, Total	EPA 200.7 Rev. 4.4
	EPA 6010C
	EPA 6020A
	EPA 200.8 Rev. 5.4
Vanadium, Total	EPA 200.7 Rev. 4.4
	EPA 6010C
	EPA 6020A
	EPA 200.8 Rev. 5.4
Zinc, Total	EPA 200.7 Rev. 4.4
	EPA 6010C
	EPA 6020A
	EPA 200.8 Rev. 5.4

Metals III

Cobalt, Total	EPA 200.7 Rev. 4.4
	EPA 6010C
	EPA 6020A
	EPA 200.8 Rev. 5.4
Molybdenum, Total	EPA 200.7 Rev. 4.4
	EPA 6010C

Metals III

Molybdenum, Total	EPA 6020A
	EPA 200.8 Rev. 5.4
Palladium, Total	EPA 6020A
	EPA 200.8 Rev. 5.4
Platinum, Total	EPA 6020A
	EPA 200.8 Rev. 5.4
Thallium, Total	EPA 200.7 Rev. 4.4
	EPA 6010C
	EPA 6020A
	EPA 200.8 Rev. 5.4
Tin, Total	EPA 200.7 Rev. 4.4
	EPA 6010C
	EPA 6020A
	EPA 200.8 Rev. 5.4
Titanium, Total	EPA 200.7 Rev. 4.4
	EPA 6010C
	EPA 6020A
	EPA 200.8 Rev. 5.4
Uranium (Mass)	EPA 6020A
	EPA 200.8 Rev. 5.4

Mineral

Hardness, Total	EPA 200.7 Rev. 4.4
	SM 2340B-97,-11

Miscellaneous

Boron, Total	EPA 200.7 Rev. 4.4

Serial No.: 56114

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MS. SARAH CHERNEY
PACE ANALYTICAL SERVICES, LLC - MINNEAPOLIS MN
1700 ELM STREET SE SUITE 200
MINNEAPOLIS, MN 55414-2485

NY Lab Id No: 11647

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Miscellaneous		**Polychlorinated Biphenyls**	
Boron, Total	EPA 6010C	PCB 133	EPA 1668 A
	EPA 6020A	PCB 136	EPA 1668 A
	EPA 200.8 Rev. 5.4	PCB 137	EPA 1668 A
		PCB 14	EPA 1668 A
Polychlorinated Biphenyls		PCB 141	EPA 1668 A
PCB 1	EPA 1668 A	PCB 142	EPA 1668 A
PCB 10	EPA 1668 A	PCB 144	EPA 1668 A
PCB 103	EPA 1668 A	PCB 145	EPA 1668 A
PCB 104	EPA 1668 A	PCB 146	EPA 1668 A
PCB 105	EPA 1668 A	PCB 148	EPA 1668 A
PCB 106	EPA 1668 A	PCB 15	EPA 1668 A
PCB 109	EPA 1668 A	PCB 150	EPA 1668 A
PCB 11	EPA 1668 A	PCB 152	EPA 1668 A
PCB 111	EPA 1668 A	PCB 154	EPA 1668 A
PCB 112	EPA 1668 A	PCB 155	EPA 1668 A
PCB 114	EPA 1668 A	PCB 156	EPA 1668 A
PCB 118	EPA 1668 A	PCB 158	EPA 1668 A
PCB 120	EPA 1668 A	PCB 159	EPA 1668 A
PCB 121	EPA 1668 A	PCB 16	EPA 1668 A
PCB 122	EPA 1668 A	PCB 160	EPA 1668 A
PCB 123	EPA 1668 A	PCB 161	EPA 1668 A
PCB 126	EPA 1668 A	PCB 162	EPA 1668 A
PCB 127	EPA 1668 A	PCB 165	EPA 1668 A
PCB 130	EPA 1668 A	PCB 167	EPA 1668 A
PCB 131	EPA 1668 A	PCB 169	EPA 1668 A
PCB 132	EPA 1668 A	PCB 17	EPA 1668 A

Serial No.: 56114

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

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CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

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All approved analytes are listed below:

Polychlorinated Biphenyls		Polychlorinated Biphenyls	
PCB 170	EPA 1668 A	PCB 207	EPA 1668 A
PCB 172	EPA 1668 A	PCB 208	EPA 1668 A
PCB 174	EPA 1668 A	PCB 209	EPA 1668 A
PCB 175	EPA 1668 A	PCB 22	EPA 1668 A
PCB 176	EPA 1668 A	PCB 23	EPA 1668 A
PCB 177	EPA 1668 A	PCB 24	EPA 1668 A
PCB 178	EPA 1668 A	PCB 25	EPA 1668 A
PCB 179	EPA 1668 A	PCB 27	EPA 1668 A
PCB 181	EPA 1668 A	PCB 3	EPA 1668 A
PCB 182	EPA 1668 A	PCB 31	EPA 1668 A
PCB 184	EPA 1668 A	PCB 32	EPA 1668 A
PCB 186	EPA 1668 A	PCB 34	EPA 1668 A
PCB 188	EPA 1668 A	PCB 35	EPA 1668 A
PCB 189	EPA 1668 A	PCB 36	EPA 1668 A
PCB 19	EPA 1668 A	PCB 37	EPA 1668 A
PCB 190	EPA 1668 A	PCB 38	EPA 1668 A
PCB 191	EPA 1668 A	PCB 39	EPA 1668 A
PCB 192	EPA 1668 A	PCB 4	EPA 1668 A
PCB 195	EPA 1668 A	PCB 46	EPA 1668 A
PCB 2	EPA 1668 A	PCB 48	EPA 1668 A
PCB 201	EPA 1668 A	PCB 5	EPA 1668 A
PCB 202	EPA 1668 A	PCB 52	EPA 1668 A
PCB 203	EPA 1668 A	PCB 54	EPA 1668 A
PCB 204	EPA 1668 A	PCB 55	EPA 1668 A
PCB 205	EPA 1668 A	PCB 57	EPA 1668 A
PCB 206	EPA 1668 A	PCB 58	EPA 1668 A

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All approved analytes are listed below:

Polychlorinated Biphenyls	
PCB 6	EPA 1668 A
PCB 60	EPA 1668 A
PCB 63	EPA 1668 A
PCB 64	EPA 1668 A
PCB 66	EPA 1668 A
PCB 67	EPA 1668 A
PCB 68	EPA 1668 A
PCB 7	EPA 1668 A
PCB 72	EPA 1668 A
PCB 77	EPA 1668 A
PCB 78	EPA 1668 A
PCB 79	EPA 1668 A
PCB 8	EPA 1668 A
PCB 80	EPA 1668 A
PCB 81	EPA 1668 A
PCB 82	EPA 1668 A
PCB 83	EPA 1668 A
PCB 89	EPA 1668 A
PCB 9	EPA 1668 A
PCB 92	EPA 1668 A
PCB 94	EPA 1668 A
PCB 95	EPA 1668 A
PCB 96	EPA 1668 A
PCB 99	EPA 1668 A
PCB-1016	EPA 8082A
PCB-1221	EPA 8082A

Polychlorinated Biphenyls	
PCB-1232	EPA 8082A
PCB-1242	EPA 8082A
PCB-1248	EPA 8082A
PCB-1254	EPA 8082A
PCB-1260	EPA 8082A
Residue	
Solids, Total Suspended	SM 2540 D-97,-11
Sample Preparation Methods	
	EPA 3010A
	EPA 3510C
	EPA 3020A

Serial No.: 56114

NELAP RECOGNIZED TNI ACCREDITATION BODY

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All approved analytes are listed below:

Characteristic Testing

Synthetic Precipitation Leaching Proc.	EPA 1312
TCLP	EPA 1311

Dioxins and Furans

1,2,3,4,6,7,8,9-Octachlorodibenzofuran	EPA 8290A
1,2,3,4,6,7,8,9-Octachlorodibenzo-p-diox	EPA 8290A
1,2,3,4,6,7,8-Heptachlorodibenzofuran	EPA 8290A
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxi	EPA 8290A
1,2,3,4,7,8,9-Heptachlorodibenzofuran	EPA 8290A
1,2,3,4,7,8-Hexachlorodibenzofuran	EPA 8290A
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	EPA 8290A
1,2,3,6,7,8-Hexachlorodibenzofuran	EPA 8290A
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	EPA 8290A
1,2,3,7,8,9-Hexachlorodibenzofuran	EPA 8290A
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	EPA 8290A
1,2,3,7,8-Pentachlorodibenzofuran	EPA 8290A
1,2,3,7,8-Pentachlorodibenzo-p-dioxin	EPA 8290A
2,3,4,6,7,8-Hexachlorodibenzofuran	EPA 8290A
2,3,4,7,8-Pentachlorodibenzofuran	EPA 8290A
2,3,7,8-Tetrachlorodibenzofuran	EPA 8290A
2,3,7,8-Tetrachlorodibenzo-p-dioxin	EPA 8290A

Metals I

Barium, Total	EPA 6010C
	EPA 6020A
Cadmium, Total	EPA 6010C
	EPA 6020A

Metals I

Calcium, Total	EPA 6010C
	EPA 6020A
Chromium, Total	EPA 6010C
	EPA 6020A
Copper, Total	EPA 6010C
	EPA 6020A
Iron, Total	EPA 6010C
	EPA 6020A
Lead, Total	EPA 6010C
	EPA 6020A
Magnesium, Total	EPA 6010C
	EPA 6020A
Manganese, Total	EPA 6010C
	EPA 6020A
Nickel, Total	EPA 6010C
	EPA 6020A
Potassium, Total	EPA 6010C
	EPA 6020A
Silver, Total	EPA 6010C
	EPA 6020A
Sodium, Total	EPA 6010C
	EPA 6020A
Strontium, Total	EPA 6020A

Metals II

Aluminum, Total	EPA 6010C

Serial No.: 56115

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Metals II	
Aluminum, Total	EPA 6020A
Antimony, Total	EPA 6010C
	EPA 6020A
Arsenic, Total	EPA 6010C
	EPA 6020A
Beryllium, Total	EPA 6010C
	EPA 6020A
Lithium, Total	EPA 6020A
Mercury, Total	EPA 7471B
Selenium, Total	EPA 6010C
	EPA 6020A
Vanadium, Total	EPA 6010C
	EPA 6020A
Zinc, Total	EPA 6010C
	EPA 6020A
Metals III	
Cobalt, Total	EPA 6010C
	EPA 6020A
Molybdenum, Total	EPA 6010C
	EPA 6020A
Thallium, Total	EPA 6010C
	EPA 6020A
Tin, Total	EPA 6010C
	EPA 6020A
Titanium, Total	EPA 6010C

Metals III	
Titanium, Total	EPA 6020A
Miscellaneous	
Boron, Total	EPA 6010C
	EPA 6020A
Polychlorinated Biphenyls	
PCB 1	EPA 1668 A
PCB 10	EPA 1668 A
PCB 103	EPA 1668 A
PCB 104	EPA 1668 A
PCB 105	EPA 1668 A
PCB 106	EPA 1668 A
PCB 109	EPA 1668 A
PCB 11	EPA 1668 A
PCB 111	EPA 1668 A
PCB 112	EPA 1668 A
PCB 114	EPA 1668 A
PCB 118	EPA 1668 A
PCB 120	EPA 1668 A
PCB 121	EPA 1668 A
PCB 122	EPA 1668 A
PCB 123	EPA 1668 A
PCB 126	EPA 1668 A
PCB 127	EPA 1668 A
PCB 130	EPA 1668 A
PCB 131	EPA 1668 A

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All approved analytes are listed below:

Polychlorinated Biphenyls		Polychlorinated Biphenyls	
PCB 132	EPA 1668 A	PCB 167	EPA 1668 A
PCB 133	EPA 1668 A	PCB 169	EPA 1668 A
PCB 136	EPA 1668 A	PCB 17	EPA 1668 A
PCB 137	EPA 1668 A	PCB 170	EPA 1668 A
PCB 14	EPA 1668 A	PCB 172	EPA 1668 A
PCB 141	EPA 1668 A	PCB 174	EPA 1668 A
PCB 142	EPA 1668 A	PCB 175	EPA 1668 A
PCB 144	EPA 1668 A	PCB 176	EPA 1668 A
PCB 145	EPA 1668 A	PCB 177	EPA 1668 A
PCB 146	EPA 1668 A	PCB 178	EPA 1668 A
PCB 148	EPA 1668 A	PCB 179	EPA 1668 A
PCB 15	EPA 1668 A	PCB 181	EPA 1668 A
PCB 150	EPA 1668 A	PCB 182	EPA 1668 A
PCB 152	EPA 1668 A	PCB 184	EPA 1668 A
PCB 154	EPA 1668 A	PCB 186	EPA 1668 A
PCB 155	EPA 1668 A	PCB 188	EPA 1668 A
PCB 156	EPA 1668 A	PCB 189	EPA 1668 A
PCB 157	EPA 1668 A	PCB 19	EPA 1668 A
PCB 158	EPA 1668 A	PCB 190	EPA 1668 A
PCB 159	EPA 1668 A	PCB 191	EPA 1668 A
PCB 16	EPA 1668 A	PCB 192	EPA 1668 A
PCB 160	EPA 1668 A	PCB 194	EPA 1668 A
PCB 161	EPA 1668 A	PCB 195	EPA 1668 A
PCB 162	EPA 1668 A	PCB 196	EPA 1668 A
PCB 164	EPA 1668 A	PCB 2	EPA 1668 A
PCB 165	EPA 1668 A	PCB 201	EPA 1668 A

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All approved analytes are listed below:

Polychlorinated Biphenyls		Polychlorinated Biphenyls	
PCB 202	EPA 1668 A	PCB 5	EPA 1668 A
PCB 203	EPA 1668 A	PCB 52	EPA 1668 A
PCB 204	EPA 1668 A	PCB 54	EPA 1668 A
PCB 205	EPA 1668 A	PCB 55	EPA 1668 A
PCB 206	EPA 1668 A	PCB 56	EPA 1668 A
PCB 207	EPA 1668 A	PCB 57	EPA 1668 A
PCB 208	EPA 1668 A	PCB 58	EPA 1668 A
PCB 209	EPA 1668 A	PCB 6	EPA 1668 A
PCB 22	EPA 1668 A	PCB 60	EPA 1668 A
PCB 23	EPA 1668 A	PCB 63	EPA 1668 A
PCB 24	EPA 1668 A	PCB 64	EPA 1668 A
PCB 25	EPA 1668 A	PCB 66	EPA 1668 A
PCB 27	EPA 1668 A	PCB 67	EPA 1668 A
PCB 3	EPA 1668 A	PCB 68	EPA 1668 A
PCB 31	EPA 1668 A	PCB 7	EPA 1668 A
PCB 32	EPA 1668 A	PCB 72	EPA 1668 A
PCB 34	EPA 1668 A	PCB 77	EPA 1668 A
PCB 35	EPA 1668 A	PCB 78	EPA 1668 A
PCB 36	EPA 1668 A	PCB 79	EPA 1668 A
PCB 37	EPA 1668 A	PCB 8	EPA 1668 A
PCB 38	EPA 1668 A	PCB 80	EPA 1668 A
PCB 39	EPA 1668 A	PCB 81	EPA 1668 A
PCB 4	EPA 1668 A	PCB 82	EPA 1668 A
PCB 42	EPA 1668 A	PCB 83	EPA 1668 A
PCB 46	EPA 1668 A	PCB 84	EPA 1668 A
PCB 48	EPA 1668 A	PCB 89	EPA 1668 A

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All approved analytes are listed below:

Polychlorinated Biphenyls

PCB 9	EPA 1668 A
PCB 92	EPA 1668 A
PCB 94	EPA 1668 A
PCB 95	EPA 1668 A
PCB 96	EPA 1668 A
PCB 99	EPA 1668 A
PCB-1016	EPA 8082A
PCB-1221	EPA 8082A
PCB-1232	EPA 8082A
PCB-1242	EPA 8082A
PCB-1248	EPA 8082A
PCB-1254	EPA 8082A
PCB-1260	EPA 8082A

Sample Preparation Methods

	EPA 3050B
	EPA 3550C
	EPA 3540C
	EPA 3546

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All approved analytes are listed below:

Chlorinated Hydrocarbons	
1,2,4-Trichlorobenzene	EPA TO-14A
	EPA TO-15
Hexachlorobutadiene	EPA TO-14A
	EPA TO-15
Polychlorinated Biphenyls	
PCBs and Aroclors	EPA TO-10A
	EPA TO-4A
Polynuclear Aromatics	
Acenaphthene	EPA TO-13A
Acenaphthylene	EPA TO-13A
Anthracene	EPA TO-13A
Benzo(a)anthracene	EPA TO-13A
Benzo(a)pyrene	EPA TO-13A
Benzo(b)fluoranthene	EPA TO-13A
Benzo(ghi)perylene	EPA TO-13A
Benzo(k)fluoranthene	EPA TO-13A
Chrysene	EPA TO-13A
Dibenzo(a,h)anthracene	EPA TO-13A
Fluoranthene	EPA TO-13A
Fluorene	EPA TO-13A
Indeno(1,2,3-cd)pyrene	EPA TO-13A
Naphthalene	EPA TO-13A
	EPA TO-15
Phenanthrene	EPA TO-13A
Pyrene	EPA TO-13A

Purgeable Aromatics	
1,2,4-Trimethylbenzene	EPA TO-14A
	EPA TO-15
1,2-Dichlorobenzene	EPA TO-14A
	EPA TO-15
1,3,5-Trimethylbenzene	EPA TO-15
1,3-Dichlorobenzene	EPA TO-14A
	EPA TO-15
1,4-Dichlorobenzene	EPA TO-14A
	EPA TO-15
Benzene	EPA TO-14A
	EPA TO-15
	EPA TO-3
Chlorobenzene	EPA TO-14A
	EPA TO-15
Ethyl benzene	EPA TO-14A
	EPA TO-15
m/p-Xylenes	EPA TO-15
o-Xylene	EPA TO-15
Styrene	EPA TO-14A
	EPA TO-15
Toluene	EPA TO-14A
	EPA TO-15
Total Xylenes	EPA TO-14A
	EPA TO-15

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Purgeable Halocarbons	
1,1,1-Trichloroethane	EPA TO-14A
	EPA TO-15
1,1,2,2-Tetrachloroethane	EPA TO-14A
	EPA TO-15
1,1,2-Trichloro-1,2,2-Trifluoroethane	EPA TO-14A
	EPA TO-15
1,1,2-Trichloroethane	EPA TO-14A
	EPA TO-15
1,1-Dichloroethane	EPA TO-14A
	EPA TO-15
1,1-Dichloroethene	EPA TO-14A
	EPA TO-15
1,2-Dibromoethane	EPA TO-14A
	EPA TO-15
1,2-Dichloroethane	EPA TO-14A
	EPA TO-15
1,2-Dichloropropane	EPA TO-14A
	EPA TO-15
Bromodichloromethane	EPA TO-15
Bromoform	EPA TO-15
Bromomethane	EPA TO-14A
	EPA TO-15
Carbon tetrachloride	EPA TO-14A
	EPA TO-15
Chloroethane	EPA TO-14A
	EPA TO-15

Purgeable Halocarbons	
Chloroform	EPA TO-14A
	EPA TO-15
Chloromethane	EPA TO-15
cis-1,2-Dichloroethene	EPA TO-14A
	EPA TO-15
cis-1,3-Dichloropropene	EPA TO-14A
	EPA TO-15
Dibromochloromethane	EPA TO-15
Dichlorodifluoromethane	EPA TO-14A
	EPA TO-15
Methylene chloride	EPA TO-14A
	EPA TO-15
Tetrachloroethene	EPA TO-14A
	EPA TO-15
trans-1,2-Dichloroethene	EPA TO-14A
	EPA TO-15
trans-1,3-Dichloropropene	EPA TO-14A
	EPA TO-15
Trichloroethene	EPA TO-14A
	EPA TO-15
Trichlorofluoromethane	EPA TO-14A
	EPA TO-15
Vinyl chloride	EPA TO-14A
	EPA TO-15

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Volatile Chlorinated Organics	
Benzyl chloride	EPA TO-14A
	EPA TO-15
Volatile Organics	
1,2-Dichlorotetrafluoroethane	EPA TO-14A
	EPA TO-15
1,3-Butadiene	EPA TO-15
2-Butanone (Methylethyl ketone)	EPA TO-15
4-Methyl-2-Pentanone	EPA TO-15
Acetone	EPA TO-15
Carbon Disulfide	EPA TO-15
Cyclohexane	EPA TO-15
Hexane	EPA TO-15
Isopropanol	EPA TO-15
Methyl tert-butyl ether	EPA TO-15
n-Heptane	EPA TO-15
Vinyl acetate	EPA TO-15

Serial No.: 56116

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ATTACHMENT 2

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STANDARD OPERATING PROCEDURE

MEASUREMENT OF SOLIDS IN WATER AND WASTEWATER

Reference Methods: SM 2540-B, -C, and –D and EPA 160.4

Local SOP Number:	S-MN-I-528-Rev.15
Effective Date:	Date of Final Signature
Supersedes:	S-MN-I-528-Rev.14

APPROVALS

Laboratory General Manager — Date: 06 JUN 2016

Laboratory Quality Manager — Date: 06 JUN 2016

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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TABLE OF CONTENTS

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1. Purpose/Identification Of Method

1.1. This Standard Operating Procedure (SOP) describes operations used to measure Total Solids (TS), Total Suspended Solids (TSS), Total Dissolved Solids (TDS), and Total Volatile Solids (TVS) in water samples, and TS and TVS on solids, based on Standard Methods 2540-B, 2540-C, and 2540-D and EPA 160.4.

2. Summary of Method

2.1. Total Solids (TS) – A well-mixed sample is evaporated to dryness and the residue solids are measured gravimetrically.

2.2. Total Suspended Solids (TSS) – A well-mixed sample is filtered. The residue collected by the filter is dried and measured gravimetrically.

2.3. Total Dissolved Solids (TDS) – A well-mixed sample is filtered. The filtrate passing through the filter is evaporated to dryness and the residual solids is measured gravimetrically.

2.4. Total Volatile Solids (TVS, TVSS, or TVDS) – Residue obtained from the determination of TS, TSS, or TDS is ignited at 550 ºC in a muffle furnace. The loss of weight on ignition is reported as mg/L volatile solid (TVS if the result was obtained from ashing a TS sample, TVSS if the result was from a TSS sample, or TVDS if the result was obtain from a TDS sample).

2.5. Solid matrices are reported as a percentage for TS and TVS.

3. Scope and Application

3.1. Personnel: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method.

3.2. Parameters: This SOP applies to matter suspended or dissolved in water or wastewater.

4. Applicable Matrices

4.1. This SOP is applicable to water samples, including drinking water, groundwater, municipal and industrial wastewater.

5. Limits Of Detection and Quantitation

5.1. The reporting limit (LOQ) for all analytes is 10 mg/L for a default volume of 100 mL. All current limits are listed in the LIMS and are available by request from the Quality Manager.

6. Interferences

6.1. Non-representative materials, e.g., leaves and sticks should be removed from the sample prior to measurement unless it is determined that their inclusion is desired. If floating oil and grease are present, the sample should be dispersed by blending prior to analysis.

6.2. Measurements are subject to negative bias for samples containing significant quantities of ammonium carbonate, volatile organics, or other volatile materials that could be lost during drying.

6.3. The residue of samples for TS and TDS that are highly mineralized, especially containing significant concentrations of calcium, magnesium, chloride, and/or sulfate may be hydroscopic and will require longer drying, desiccation, and rapid weighing.

6.4. Samples for TS and TDS containing high concentrations of bicarbonate will require careful, and possibly prolonged, drying to ensure that all bicarbonate is converted to carbonate.

6.5. The volumes of aliquots for TS and TDS should be selected to limit the total residue to 200 mg to prevent the residue from crusting over and trapping water during drying.

6.6. Samples for TSS with high TDS, such as saline waters, brines, and some wastes, may be subject to positive bias. Care must be taken to properly rinse the filter to minimize the bias.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Collection, Preservation, Storage and Holding Time Table

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	Samples are collected in plastic bottles	N/A	Samples are stored above freezing but below 6°C	Analyze as soon as possible to minimize microbiological decomposition of organic solids, holding time not to exceed 7 days from collection.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

9. Equipment and Supplies (Including Computer Hardware And Software)

9.1. Table 9.1 Equipment and Supplies Table

Supply	Description	Vendor/ Item # / Description
Analytical Balance	Electronic with RS-232 output, capable of weighing 0.0001g	Mettler Toledo, AB135-S
Drying Oven	Capable of maintaining temperature at 103-105°C for TSS, capable of holding temperature at 178-182°C for TDS	VWR 1370F, Precision Scientific
Muffle Furnace	Capable of maintaining temperature at 550°C for volatile solids	Fisher Scientific, or equivalent
Vacuum Filtration System	Including filter holder, membrane filter funnel, vacuum flask and vacuum pump	Pall
EPIC Horizon (LIMS)	Data Reporting Software	See master list for current version
LIMSLink	Data Transmission Software	See master list for current version
Microsoft Excel	Spreadsheet software	See master list for current version
Dessicator	General laboratory equipment	Labconco
Indicating Dessicant	General laboratory equipment	Drierite, 23005
Non-indicating Dessicant	General laboratory equipment	Drierite, 13005
Ceramic Evaporating Dishes (Crucibles)	For use in TVS	Fisher Scientific, or equivalent
Beaker	200 mL capacity, tall form. For use in TDS and TS.	Fisher Scientific part # 02-546B, or equivalent
Glass Fiber Filters	Pre-washed and dried. For use in TDS.	Environmental Express part # F92447MM
Glass Fiber Filters	Pre-washed, dried, pre-weighed and barcoded. For use in TSS.	Environmental Express part # F93447MM
Wash Bottle	Nalgene, one piece stem or equivalent	Fisher Scientific, or equivalent

Glass Fiber Filter (TVSS)	Pre-washed and dried	Environmental Express part # F92447VOL
Glass Fiber Filters (Sand Filtrate)	General laboratory equipment	Millipore part # AP2504700
Aluminum Dish	70 mL. Low form weighing dish, smooth.	Fisher part # 08-732-103

10. Reagents and Standards

10.1. Table 10.1 - Reagents and Standards Table

Reagent/Standard	Concentration/ Description	Requirements/ Vendor/ Item #
De-Ionized (DI) Water		Verify daily that pH and specific conductivity are within acceptable limits.
CeLite Filter Aid	TSS dry standard. Diatomaceous earth. Store at room temperature. Expires per manufacturer's specifications or five years after opening, whichever is sooner.	Fisher part # C212-500 or equivalent
Sodium Chloride (NaCl)	TDS dry standard. Store at room temperature. Expires per manufacturer's specifications or five years after opening, whichever is sooner.	Fisher part # S640-500 or equivalent
TS/TSS/TDS Standard	Purchased premade. Store at room temperature. Expires per manufacturer's specifications. TDS = 1000 mg/L, TSS = 100 mg/L, TS = 1100 mg/L	Environmental Express part # NS1QC1-55H or equivalent
Hydrochloric Acid (HCl)	6N HCl. For use in cleaning glassware. Store at room temperature. Expires per manufacturer's specifications or one year after opening, whichever is sooner.	Fisher part # 3750.1-1 or equivalent
Solids QR Sample	For use in training new employees on TSS/TDS. See 13.2.2.	TSS: ERA part # 507QR or equivalent TDS: ERA part # 506QR or equivalent

10.2. Table 10.2 - Working Standard Dilutions and Concentrations

Standard	Standard(s) Used	Standard(s) Amount	Solvent	Final Total Volume	Final Concentration
TSS Standard	CeLite Filter Aid	0.2 g	DI Water	2000 mL	100 mg/L
TDS Standard	NaCl	2 g	DI Water	2000 mL	1000 mg/L
TSS/TDS/TS Combined Standard	CeLite Filter Aid	0.2 g	DI Water	2000 mL	100 mg TSS/L 1000 mg TDS/L 1100 mg TS/L
	NaCl	2 g			
10% HCl	HCl	100 mL	DI Water	1000 mL	10% HCl

10.2.1. All standards have a six month expiration date. Store at room temperature.

11. Calibration and Standardization

11.1. Daily calibration of the balance is required following SOP S-MN-Q-264 – Support Equipment (or equivalent replacement). Record in associated balance calibration logbook. Calibration limits are found in the balance calibration logbook; if values exceed these limits, recalibrate the balance.

11.2. All balances must be certified by an outside agency on an annual basis with documentation of the calibration maintained in the QA office.

11.3. Establishing Constant Weight

11.3.1. Weigh the vessel (filter pad, beaker or crucible) and dry in an oven for at least 1 hour (104 ± 1ºC for TSS filter pads and TS beakers or crucibles, 180 ± 2ºC for TDS beakers, 550ºC muffle furnace for TVSS filter pads and TVS crucibles).

11.3.2. Remove from the oven and allow them to cool to room temperature in a desiccator.

11.3.2.1. If done in a room with < 20% humidity, the vessels may be allowed to cool on a benchtop for approximately 15 minutes to allow more rapid cooling.

11.3.3. Weigh the filter pad, beaker or crucible again. If the second weight is within ±0.5 mg of the first, constant weight is established and analysis is complete, otherwise repeat 11.3.1 through 11.3.3 for a third weight and, if necessary, a fourth weight.

11.3.3.1. 4% Rule (only applicable to residue, does not apply to clean vessel weights): If any weight measurement agrees within 4% of a previous weight, it is considered a constant weight and analysis is complete.

11.3.3.2. If the fourth weight is still not within ±0.5 mg, use the fourth weight and qualify the sample data as not having established constant weight.

11.3.3.3. TSS filters may be purchased pre-weighed and may be used immediately. One filter per manufacturer's lot must be weighed to verify correctness.

11.3.3.4. All weights must be recorded and the date/time and oven temperature must be recorded in the electronic prep log.

12. Procedure

12.1. Total Solids (TS)

12.1.1. Wash beakers (or crucibles if TVS is being performed) with phosphate-free soap and warm tap water. Rinse three times or until free of soap. Rinse three times with DI water and dry at 180±2°C for at least 1 hour.

12.1.2. Assign clean, dry beakers to each sample and record the tare weight to the nearest 0.1 mg using the constant weight procedure described in section 11.3.

12.1.3. Choose a sample volume to yield a residue of at least 25 mg but less than 200 mg. If there is not a reliable indication of solids content, choose 100 mL if the sample appears clean or, as little as 10 mL if the sample appears to have solids.

12.1.4. Shake thoroughly to homogenize the sample and measure the chosen aliquot volume in a graduated cylinder and pour into the respective beaker.

12.1.5. Rinse the graduated cylinder with ~10 mL DI water and pour into the beaker. Repeat this step twice. All three rinses should be poured into the beaker as part of the analysis.

12.1.6. Optional: Evaporate the beakers in an oven no higher than 105ºC to complete dryness, preferably overnight.

12.1.7. Place the evaporated beakers in an oven at 104 ± 1ºC for at least 1 hour.

12.1.8. Remove the beakers from the oven and allow them to cool to room temperature in a desiccator.

12.1.8.1. If done in a room with < 20% humidity, the beakers may be allowed to cool on a benchtop for approximately 5 minutes to allow more rapid cooling.

12.1.9. After cooling to room temperature, weigh each beaker to the nearest 0.1 mg.

12.1.10. Repeat steps 12.1.7 through 12.1.9 and establish constant weight as described in section 11.3.

12.1.11. Record all weights, dates and times of oven and desiccator tracking as well as oven temperatures in the electronic prep log (F-MN-I-318 for TS, F-MN-I-375 for beaker preweights).

12.2. Total Suspended Solids (TSS) & Sand Filtrate

12.2.1. Use ProWeigh filter pads (prewashed and preweighed) from Environmental Express. Verify the weight indicated on the weigh pan or record the actual weight if it differs from the vendor indicated weight.

12.2.2. For sand filtrate, TVSS or for TSS when ProWeigh filter pads are not available, identify and wash each filter pad with three 20 mL aliquots of DI water and vacuum to dryness between each aliquot. Establish constant weight for each filter pad as described in section 11.3.

12.2.2.1. Use Millipore part # AP2504700 for sand filtrate.

12.2.2.2. Use Environmental Express part # F92447VOL for TVSS.

12.2.2.3. Use Environmental Express part # F92447MM for TSS when ProWeigh filter pads are not available.

12.2.3. Choose a sample volume to yield a residue of at least 1 mg but less than 200 mg.

12.2.3.1. For TSS low level, filter a 500 mL volume if the sample appears clean.

12.2.3.1.1. Per client request, filter a 1000 mL volume if the sample is drinking water or other very clean surface water.

12.2.3.2. For wastewaters, choose 100 mL if the sample appears clean. When filtering less than 10 mL, dilute the aliquot of sample in at least 10 mL DI water prior to filtering in order to disperse the sample on the filter pad evenly.

12.2.3.3. For sand filtrate, filter a 1000 mL volume.

12.2.4. Place a clean filter pad on the filtration manifold, handling the filter pad with forceps only. Wet the filter with a small volume of DI water to seat it. Discard the rinsates if the sample is also being analyzed for TDS.

12.2.5. Shake the sample thoroughly and measure the chosen aliquot volume using a class A graduated cylinder.

12.2.6. Filter the sample. Rinse the graduated cylinder and wash the filter three times with approximately 10 mL of DI water. Once filtration is complete, maintain the filter vacuum for about three minutes and until filter has come to complete dryness.

12.2.6.1. If the sample is also being analyzed for TDS, retain the filtrate. Transfer the filtrate to an evaporating vessel and complete the analysis for TDS as described in Section 12.3.

12.2.6.2. If total volatile suspended solids (TVSS) are also to be performed, transfer the filter pad to a ceramic evaporating dish and complete the analysis for TVSS as described in Section 12.4.

12.2.7. Remove the filter pad with the forceps and place it back into its respective aluminum pan.

12.2.7.1. If the sample contains oils, solvents, surfactants, dyes and other organic materials that adhere strongly to surfaces, wash all parts of the filter apparatus as well as the graduated cylinder with hot, soapy water and a brush until all solids are thoroughly removed. Failure to do so may result in cross-contamination between samples.

12.2.8. Place the pans with filter pads onto drying racks in oven at 104±1ºC for at least 1 hour.

12.2.9. Remove the samples from the oven and place them in a desiccator to cool.

12.2.10. After cooling to room temperature, weigh each filter pad to the nearest 0.1 mg.

12.2.11. Repeat steps 12.2.8 through 12.2.10 and establish constant weight as described in section 11.3.

12.2.12. Record all weights, dates and times of oven and desiccator tracking as well as oven temperatures in the electronic prep log (F-MN-I-325 for TSS, F-MN-I-375 for sand filter preweights).

12.3. Total Dissolved Solids (TDS)

12.3.1. Wash beakers (or crucibles if TVDS is being performed) with phosphate-free soap and warm tap water. Rinse three times or until free of soap. Rinse three times with DI water and dry at 180 ± 2°C for at least 1 hour.

12.3.2. Assign clean, dry beakers to each sample and record the tare weight to the nearest 0.1 mg using the constant weight procedure described in section 11.3.

12.3.3. Choose a sample volume to yield a residue of at least 25 mg but less than 200 mg. Choose 100 mL by default but if sample is suspected of having large amounts of TDS, proceed to 12.3.3.1 to prescreen. When filtering less than 10 mL, dilute the aliquot of sample in at least 10 mL DI water prior to filtering in order to evenly disperse the sample on the filter pad.

12.3.3.1. Prescreen samples to determine the general range of TDS using the Analog Total Dissolved Solids Meter.

12.3.3.2. Rinse the cell cup three times with the sample.

12.3.3.3. Fill the cell with the sample to at least ¼" above the upper electrode.

12.3.3.4. Press the black button and read the meter.

12.3.4. Prepare the filter system with a prewashed filter pad. If the sample will also be analyzed for TSS, use a ProWeigh filter pad and handle the filter with forceps only.

12.3.5. Wet the filter with a small volume of DI water to seat it and thoroughly rinse the vacuum flask with DI water. Discard the rinse water.

12.3.6. Shake the sample thoroughly and measure the chosen aliquot volume using a class A graduated cylinder.

12.3.7. Filter the sample. Rinse the graduated cylinder and wash the filter three times with approximately 10 mL of DI water. Once filtration is complete, maintain the filter vacuum for about three minutes.

12.3.8. Transfer the filtrate to its assigned beaker. Rinse the flask three times with approximately 10 mL of DI water and add the rinseates to the beaker.

12.3.8.1. If the sample is also being analyzed for TSS, transfer the filter pad to its respective pan and complete the analysis for TSS as described in Section 12.2.

12.3.8.2. If the sample contains oils, solvents, surfactants, dyes and other organic materials that adhere strongly to surfaces, wash all parts of the filter apparatus as well as the graduated cylinder with hot, soapy water and a brush until all solids are thoroughly removed. Failure to do so may result in cross-contamination between samples.

12.3.9. Optional: Evaporate the beakers in an oven no higher than 1ºC to complete dryness, preferably overnight.

12.3.10. Place the evaporated beakers in an oven at 180±2ºC for at least 1 hour.

12.3.11. Remove the beakers from the oven and allow them to cool to room temperature in a desiccator.

12.3.11.1. If done in a room with < 20% humidity, the beakers may be allowed to cool on a bench top for approximately 5 minutes to allow more rapid cooling.

12.3.12. After cooling to room temperature, weigh each beaker to the nearest 0.1 mg.

12.3.13. Repeat steps 12.3.10 through 12.3.12 and establish constant weight as described in section 11.3.

12.3.14. Record all weights, dates and times of oven and desiccator tracking as well as oven temperatures in the electronic prep log (F-MN-I-324 for TDS, F-MN-I-375 for beaker preweights).

12.4. Volatile Solids (TVS, TVDS, or TVSS)

12.4.1. Wash crucibles with phosphate-free soap and warm tap water. Rinse three times or until free of soap. Rinse at least three times with DI water and ignite in a muffle furnace at 550°C for 1 hour.

12.4.2. Assign crucibles to each sample and record the tare weight to the nearest 0.1 mg using the constant weight procedure described in section 11.3.

12.4.3. Obtain the dry residue from a TS, TDS, or TSS determination. Note: The residue for TS and TDS is obtained by drying the sample directly in a prepared crucible through the respective drying process for each analysis. The residue for TSS is obtained by filtering the sample on a pad suitable for volatization (see 12.2.2), dried through the same process for TSS samples and then placed in a clean aluminum dish for TVSS.

12.4.4. Place the crucibles (clean aluminum dishes with TSS filters if TVSS is being analyzed) into the muffle furnace at 550ºC for 30 minutes.

12.4.4.1. Note: 15 to 20 minute ignition times are usually required for a 200 mg residue; however, multiple samples and/or heavier residues may overtax the furnace and therefore necessitate a longer ignition time of 30 minutes.

12.4.5. Remove the crucibles from the furnace and cool for approximately 10 minutes. Place the crucibles in a desiccator to cool to room temperature.

12.4.6. After cooling to room temperature, weigh each beaker to the nearest 0.1 mg.

12.4.7. Repeat steps 12.4.4 through 12.4.6 and establish constant weight as described in section 11.3.

12.4.8. Record all weights, dates and times of oven and desiccator tracking as well as oven temperatures in the electronic prep log (F-MN-I-318 for TVS, F-MN-I-375 for crucible preweights).

12.5. Cleaning Glassware with HCl

12.5.1. As beakers and other glassware become heavily used, solid deposits may appear on surfaces that are impossible to remove with hot, soapy water and a brush. In this case, a solution of 10% hydrochloric acid (HCl) can be used to remove solids.

12.5.2. Prepare 10% HCl as outlined in table 10.2. Fill dirty beakers with as much solution as needed to cover any hard-to-remove solids and allow to sit for several minutes.

12.5.3. Pour out HCl from beaker and scrub with brush. Rinse beaker thoroughly and use normal glassware cleaning procedures.

12.5.4. Note: The 10% HCl solution may be re-used several times, until it has lost its efficacy.

13. Quality Control

13.1. Table 13.1 Quality Control Table

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	100 mL of DI water	Once per batch of up to 20 samples. An MB is not analyzed in TVS or TVDS.	Absolute value must be less than the reporting limit. Per QAPP or client specifications, alternate criteria such as evaluating to ½ RL may apply.	Re-analyze associated samples. ***Exceptions:*** If sample ND, report sample without qualification; If sample result >10x MB detects, report the data as it is not impacted by the blank detections; If sample result <10x MB detects and cannot be reprepared/reanalyzed, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition.
Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD)	50 mL of TSS Standard, TDS Standard or TSS/TDS/TS Combined Standard	Once per batch of up to 20 samples. An LCSD must be substituted in the event of insufficient sample volume for a duplicate sample. An LCS is not analyzed in any volatile solids	80-120% of the true value If an LCSD is analyzed, the RPD < 10% For tests undergoing volatizing by EPA 160.4, RPD ≤ 20%	Reanalyze all samples in the batch.

		analyses.		
Duplicate Sample	Client-provided sample	Once every 10 samples.	RPD ≤ 10% For tests undergoing volatizing by EPA 160.4, RPD ≤ 20%	Qualify the parent sample and duplicate with the D6 flag. If RPD is so great (>50%) that it indicates a possible lab error, reanalyze the parent sample in duplicate to confirm either the parent sample result or duplicate result. ***Exception:*** If the results of the sample and duplicate are less than 5x the RL, the data can be reported with the D8 qualifier and no further corrective action unless it is suspected that the data is in error for any other reason. If either the parent sample or the duplicate is below the MDL, RPD is not generated and no further action is necessary If either the parent sample or the duplicate is below the RL but above the MDL, qualify with a D8 flag.
Pad Weight Verification (not reported)	ProWeigh TSS pads	One pad per box of 100 (pre-weighed TSS pads) is weighed and the weight is logged on the certificate that is included in the box with the pad. This documentation is filed with the certificates of analysis for all materials received in the wetchem department.	± 0.0005 g of the weight specified by the manufacturer	If outside the acceptance criteria, attempt verification on a second pad in the lot. If the second is also outside the acceptance criteria, discard the lot.

13.2. New Solids Analyst Training

13.2.1. Follow the diagram on Attachment I for sample vessel organization when filtering and weighing back analytical batches.

13.2.2. A new analyst who is to learn one or more of the procedures described in this SOP will be audited by the staff supervisor within 30 days of his or her first day of training in solids. The audit will focus on filtering technique and will cover a few key points necessary in proper filtration as described in this SOP which include but are not limited to the following:

13.2.2.1. Seating the filter with a small amount of DI water prior to filtration.

13.2.2.2. Thoroughly homogenizing the sample prior to measuring an aliquot for filtration.

13.2.2.3. Measuring the sample aliquot using a class A graduated cylinder at eye level.

13.2.2.4. Rinsing the graduated cylinder and filter funnel at least three times with 10 mL aliquots of DI water.

13.2.2.5. Allowing the filter pad to vacuum-dry for at least three minutes after the filtration is complete or until it has come to complete dryness.

13.2.3. In addition, the analyst must successfully complete a blind QR sample within 60 days of his or her first day of training in solids. This sample will be treated the same as a proficiency testing sample.

14. Data Analysis and Calculations

14.1. See the most current Quality Manual for calculations

14.2. LCS Recovery

$$\text{LCS Recovery} = \frac{\text{SSR} \times 100\%}{\text{SA}}$$

Where:
SSR = Spike Sample Results
SA = Spike Added from spiking mix

14.3. Relative Percent Differences (RPD)

$$RPD = \frac{|A - B|}{(A + B)/2} x100$$

Where:
RPD = Relative Percent Difference
A = % Moisture for Sample
B = % Moisture for Sample Duplicate

14.4. TS, TSS and TDS

TS, TSS and TDS (mg/L) = (A-B)*1000/V
Where:
A = Weight of residue and vessel (g)
B = Tare weight of vessel (g)
V = Volume of sample aliquot (mL)

14.5. TVS, TVDS and TVSS

$$\text{TVS, TVDS or TVSS (mg/L)} = \frac{|A - B|}{V} x100$$

% TVS = (A-B)/(A) *100

Where:

A = Weight of dry sample residue and dish before ignition (mg)

B = Weight of ignited sample residue and dish (mg)

V = Volume of sample aliquot (mL)

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. See table in section 13.

16. Corrective Actions for Out-Of-Control Data

16.1. See table in section 13.

17. Contingencies for Handling Out-Of-Control or Unacceptable Data

17.1. If not specifically listed in the table in section 13, the contingencies are as follows. If there is no additional sample volume to perform re-analyses, all data will be reported as final with applicable qualifiers. If necessary, an official case narrative will be prepared by the Quality Manager or Project Manager.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. **Method Detection Limit (MDL) Study**: Not applicable to this SOP.

18.3. **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) S-ALL-Q-020 (or equivalent replacement), Training Procedures.

18.4. **Periodic performance evaluation (PE)** samples are analyzed periodically to demonstrate continuing competence per SOP S-MN-Q-258 (or equivalent replacement). Results are stored in the Quality office.

19. Method Modifications

19.1. The duplicate criteria exception of reported data outside of 10% is a modification. This is due to the fact that samples close to the reporting limit can be statistically unreliable. The 5x the RL criteria is based on the inorganic guidance provided in National Functional Guidelines.

20. Instrument/Equipment Maintenance

20.1. All maintenance activities are listed daily in maintenance logs that are assigned to each separate instrument.

21. Troubleshooting

21.1. Not applicable.

22. Safety

22.1. Standards and Reagents: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. Samples: Take precautions when handling samples. Samples should always be treated as potentially hazardous “unknowns”. The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in S-MN-S-003, Waste Handling and Management, or equivalent replacement.

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains information on pollution prevention.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.4. USEPA Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Method 160.4, Issued 1971.

25.5. Methods 2540-B, 2540-C, and 2540-D, Standard Methods for Examination of Water and Wastewater, 20th Edition (1997).

26. Tables, Diagrams, Flowcharts, and Validation Data

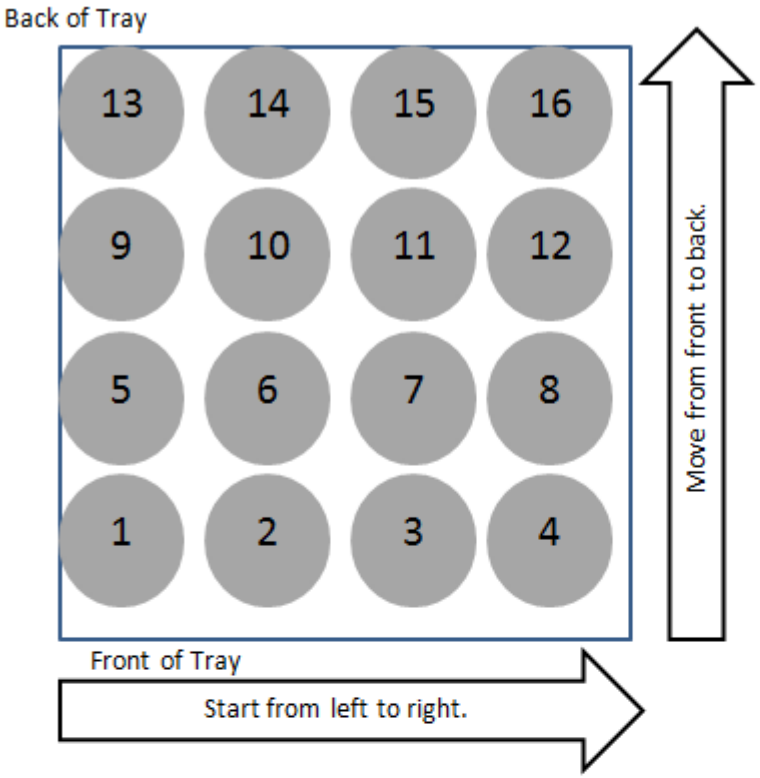
26.1. Attachment I: Filters, Beakers, and Crucibles

27. Revisions

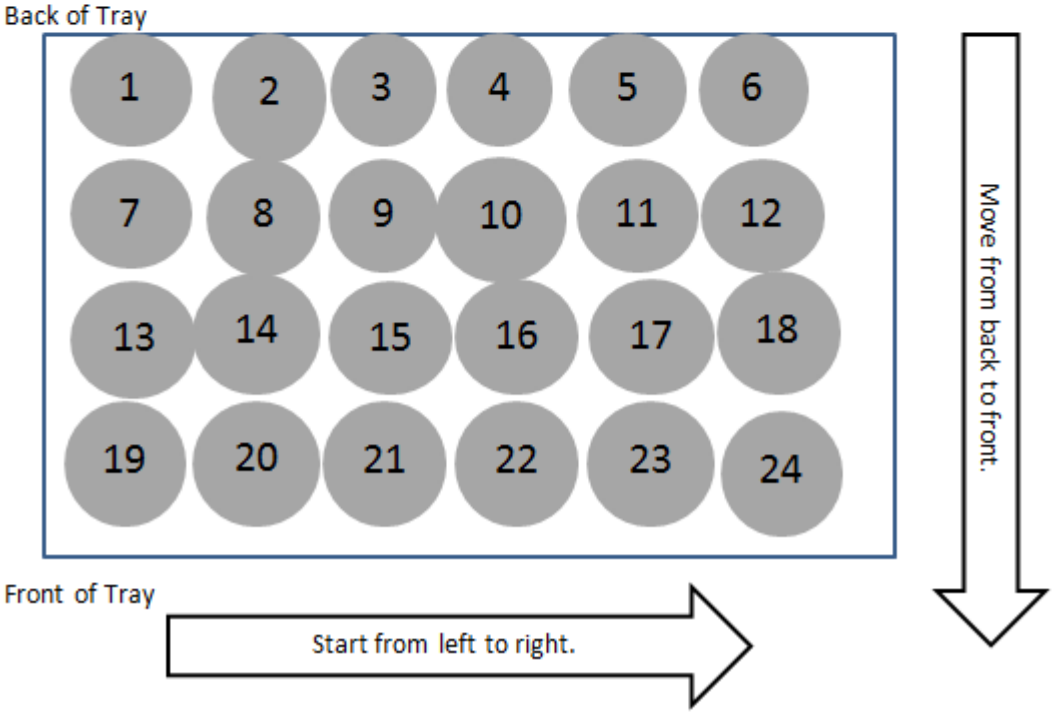
Document Number	Reason for Change	Date
S-MN-I-528-rev.15	12.1.5- Added, "All three rinses…as part of analysis." 12.3.8- Added the 3x rinse process 12.3.9- Changed 05° to 1° 13.2.1- Added directions to follow Attachment I	6/01/16

ATTACHMENT I:

Filters



Beakers/Crucibles



ATTACHMENT 3

NEW YORK STATE OF OPPORTUNITY. Department of Health

ANDREW M. CUOMO
Governor

HOWARD A. ZUCKER, M.D., J.D.
Commissioner

SALLY DRESLIN, M.S., R.N.
Executive Deputy Commissioner

LAB ID: 11411

April 01, 2017

MS. MARTHA MAIER
VISTA ANALYTICAL LABORATORY
1104 WINDFIELD WAY
EL DORADO HILLS, CA 95762

Certificate Expiration Date:
April 01, 2018

Dear Ms. Maier,

Enclosed are certificate(s) of approval issued to your environmental laboratory for the current permit year. The certificate(s) supersede(s) any previously issued one(s) and is(are) in effect through the expiration date listed. Please carefully examine the certificate(s) to insure that the categories, subcategories, analytes, and methods for which your laboratory is approved are correct. In addition, verify that your laboratory's name, address, lead technical director, and identification number are accurate.

Pursuant to NYCRR Subpart 55-2.2, original certificates must be posted conspicuously in the laboratory and copies shall be made available to any client of the laboratory upon request.

Pursuant to NYCRR Subpart 55-2.6, any misrepresentation of the fields of accreditation (category - method - analyte) for which your laboratory is approved may result in denial, suspension, or revocation of your certification. Any use of the Environmental Laboratory Approval Program (ELAP) or National Environmental Laboratory Accreditation Program (NELAP) name, reference to the laboratory's approval status, and/or using the NELAP logo in any catalogs, advertising, business solicitations, proposals, quotations, laboratory analytical reports, or other materials must include the laboratory's ELAP identification number and distinguish between testing for which the laboratory is approved and testing for which the laboratory is not approved.

If you have any questions, please contact ELAP at the New York State Department of Health (NYS DOH), Wadsworth Center, PO Box 509, Albany NY, 12201-0509; by phone at (518) 485-5570; by facsimile at (518) 485-5568; and by email at elap@health.ny.gov.

Sincerely,

Victoria Pretti

Victoria Pretti
Director and QA Officer
Environmental Laboratory Approval Program

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2018
Issued April 01, 2017

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MS. MARTHA MAIER
VISTA ANALYTICAL LABORATORY
1104 WINDFIELD WAY
EL DORADO HILLS, CA 95762

NY Lab Id No: 11411

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES POTABLE WATER
All approved analytes are listed below:

Perfluorinated Alkyl Acids

Perfluorooctanesulfonic acid (PFOS)	EPA 537
Perfluorooctanoic acid (PFOA)	EPA 537

Serial No.: 55995

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2018
Issued April 01, 2017

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EL DORADO HILLS, CA 95762

NY Lab Id No: 11411

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Dioxins and Furans

2,3,7,8-Tetrachlorodibenzofuran	EPA 1613B
2,3,7,8-Tetrachlorodibenzo-p-dioxin	EPA 613
	EPA 8280B
	EPA 1613B

Polychlorinated Biphenyls

PCB 105	EPA 1668 A
PCB 114	EPA 1668 A
PCB 118	EPA 1668 A
PCB 123	EPA 1668 A
PCB 126	EPA 1668 A
PCB 156	EPA 1668 A
PCB 157	EPA 1668 A
PCB 167	EPA 1668 A
PCB 169	EPA 1668 A
PCB 189	EPA 1668 A
PCB 77	EPA 1668 A
PCB 81	EPA 1668 A

Serial No.: 55996

Property of the New York State Department of Health. Certificates are valid only at the address shown, must be conspicuously posted, and are printed on secure paper. Continued accreditation depends on successful ongoing participation in the Program. Consumers are urged to call (518) 485-5570 to verify the laboratory's accreditation status.

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2018
Issued April 01, 2017

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MS. MARTHA MAIER
VISTA ANALYTICAL LABORATORY
1104 WINDFIELD WAY
EL DORADO HILLS, CA 95762

NY Lab Id No: 11411

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved analytes are listed below:

Dioxins and Furans	
2,3,7,8-Tetrachlorodibenzofuran	EPA 8290A
2,3,7,8-Tetrachlorodibenzo-p-dioxin	EPA 8290A
Polychlorinated Biphenyls	
PCB 105	EPA 1668 A
PCB 114	EPA 1668 A
PCB 118	EPA 1668 A
PCB 123	EPA 1668 A
PCB 126	EPA 1668 A
PCB 151	EPA 1668 A
PCB 156	EPA 1668 A
PCB 157	EPA 1668 A
PCB 167	EPA 1668 A
PCB 169	EPA 1668 A
PCB 189	EPA 1668 A
PCB 77	EPA 1668 A
PCB 81	EPA 1668 A

Serial No.: 55997

ATTACHMENT 4

SOP 31	Revision: 15	Supersedes: Rev 14
PREPARATION AND ANALYSIS OF POLYCHLORINATED BIPHENYLS (PCBS) BY METHOD 1668A/C		
Analyst Review:		
Management:		
Quality Assurance:		
Effective Date: 16 May,2017		

Revision	Description of Revision
12	Section 3.2.5: Removed Dean Stark. Section 11.1.8: Added section and CRS spiking. Section 11.1.9: Added "continue with cleanup". Sections 11.4.1 and 11.4.3: Changed sample size to 10 g. Section 12.1.1: Removed previous 12.1.1 and renumbered. Section 12.3: Revised. Section 13.2.1: Changed to 20 µL of C_{14}. Section 13.2.4 and 13.3.1: changed wiretrol to 10-20 µL. Section 13.3: Revised. Section 14.3.3: Revised MS/MSD acceptance criteria. Section 14.4.1: Revised duplicate samples acceptance criteria. Section 15.2.2: Removed PCB-28/31 valley Section 15.2.3: Added PCB-156/157 criteria. Combined Sections 15.4.5 & 15.4.6 and 15.4.7 and 15.4.8. Section 15.4.6: ICAL criteria for internal standard calibration, Table 1, 2 and 5: updated.
13	Revised language of Section 11.2
14	Added Table 9 (Quality Control Requirements).
14	No changes.
15	Revised Table 1, Table 2, Table 7 and Table 8 for the elution order of congeners PCB131, PCB133, PCB 142.

1. PURPOSE

1.1. This procedure describes the preparation and analytical techniques used for the analysis of samples for polychlorinated biphenyls (PCBs) by EPA Method 1668.

2. SCOPE

2.1. This method is for the determination of the polychlorinated biphenyl (PCB) congeners at the quantitation limits listed in Table 1 for water, soil, sediment, sludge, ash, tissue, and other sample matrices by gas chromatography/high resolution mass spectrometry/selective ion monitoring (HRGC/HRMS/SIM). The quantitation limits for this determination are provided in Table 4.

2.2. The initial calibration curve (ICAL) contains all 209 native PCB congeners. For sample analysis, those PCBs resolved as separate congeners (see Table 1) are quantified using the congener–specific relative response factor (RRF) from the ICAL. Co-eluting congeners are quantified using the RRF based on the sum of the co-eluting congeners as calculated from the ICAL.

2.3. The MDLs represent the individual congener being reported, unless the congener co-elutes with one or more other congeners. In those cases, the values represent the sum of the concentrations of the co-eluting congeners.

2.4. The reporting limits and quantitation levels are usually dependent on the level of interferences rather than instrumental limitations. The quantitation limits listed are levels at which the PCBs can be determined with only common laboratory interferences present.

2.5. Detection limits are sample-specific and congener-specific.

3. METHOD MODIFICATIONS

3.1. This method is "performance-based". Modifications to the method to overcome interferences or lower the cost of measurements are permitted, provided that all performance criteria in this method are met.

3.2. The following modifications to the method were made to improve efficiency and accuracy:

3.2.1. Vista’s initial calibration curve includes PCB congeners listed in Table 1, instead of the 27 congeners included in the method.

3.2.2. According to Section 12.2.1.1 in USEPA Method 1668, a pre-filter GMF 150 is used to remove any particles. To prevent possible contamination, Vista does not use a pre-filter in disk preparation.

3.2.3. According to Section 12.2.2.1 in the method, the sample bottle is rinsed with reagent water prior to methylene chloride. Vista rinses the sample bottle with portions of methylene chloride.

3.2.4. Under the separatory funnel extraction, the separatory funnel is rinsed with 20 mL of methylene chloride. Then the methylene chloride is placed through the sodium sulfate into the concentrator. Vista eliminated the methylene chloride rinse since sufficient rinsing of the separatory funnel is performed.

3.2.5. The method specifies that the tissue and sodium sulfate is to equilibrate for 12-14 hours prior to soxhlet extraction. Vista uses a shorter equilibration time to prevent solidification of the thimble contents.

3.2.6. In the back-extraction with base and acid, Vista uses sodium hydroxide rather than potassium hydroxide.

3.2.7. The silica gel column is not pre-eluted with methylene chloride prior to hexane.

3.2.8. In the silica gel cleanup, the container is rinsed with methylene chloride instead of hexane due to the extensive preparatory cleaning of the silica gel.

3.2.9. Vista's CS-3 standard includes the PCB congeners listed Table 1; therefore, injection of the combined 209 congener solution (15.4.2.1) is not necessary.

3.2.10. Additional internal standards are added to the solutions for more accuracy in quantitation.

3.2.11. Soil samples are kept in the refrigerator rather than freezer to ensure glass containers do not break.

3.2.12. Standard vials are not marked to detect evaporation.

3.2.13. Acid alumina cleanup may be used for coplanar analysis.

3.2.14. Tetradecane is used as a final solvent for coplanar analysis only

3.2.15. Neither corn nor other oils are used as a blank matrix for tissues.

3.2.16. The pH of aqueous samples is adjusted to 2-3.

3.2.17. High pressure filtration procedure is used for aqueous samples containing high levels of sediment/particulate based upon the

chemist's professional judgment.

3.2.18. Method 1668A requires a signal to noise ratio of 10:1 for all CB's in the initial calibration. Vista's criteria is 2.5:1 for the Di-CB's.

3.2.19. Vista monitors (m)/(m+2) for ^{13}C-HeptaCB's and (m+4)/(m+6) for the ^{13}C-nona-CB's. There are less interferences observed for these masses.

4. DEFINTIONS

4.1. Definitions are presented in Glossary.

5. CONTAMINATION AND INTERFERENCES

5.1. All materials used in the analysis shall be demonstrated to be free from interferences by running reference matrix method blanks with each sample batch.

5.2. The reference matrix should simulate, as closely as possible, the sample matrix under test. Reagent water can be used to simulate water samples; playground sand or white quartz sand can be used to simulate soils; filter paper can be used to simulate papers or similar materials.

5.3. Interferences co-extracted from samples will vary from source to source, depending on the site being sampled. The cleanup steps can be used to reduce or eliminate such interferences.

6. SAFETY

6.1. Procedures shall be carried out in a manner that protects the health and safety of all Vista employees, including the appropriate use of Personal Protective Equipment and engineering controls.

6.2. Each chemical compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level. Only highly trained personnel thoroughly familiar with handling and cautionary procedures and the associated risks should handle all compounds or reagents.

6.3. Each chemical compound should be handled in well-ventilated, controlled access laboratories.

6.4. Additional health and safety information can be obtained from Safety Data Sheet (SDS) available to all personnel involved in these analyses.

6.5. In the event of a known or potential compromise to the health and safety of a Vista associate, all work must stop and the incident reported immediately to management.

7. APPARATUS AND MATERIALS

7.1. Amber glass bottles, 1 liter & 500 mL (Teflon-lined screw cap)

7.2. Analytical Balance, capable of reading to 0.0001 g

7.3. Aquacheck and pH strips

7.4. Compaq Personal Work Station 433/500

7.5. Neslab HX200 and HX500 Water Cooler

7.6. Micromass Autospec Ultima High Resolution Mass Spectrometer (M390, M467, M504, M529, M651)

7.7. Hewlett Packard 6890 Gas Chromatograph

7.8. CTC Autosampler Model A200S

7.9. ZB-1 (Phenomenex, or equivalent) column

7.10. Drying oven, VWR Model 1320 or equivalent

7.11. Glass columns

7.12. Glass wool

7.13. Organomation 24-Station N-Evaporator

7.14. Pre-cleaned Glass fiber thimbles - coarse

7.15. Rotary evaporator

7.16. Round bottom flasks: 50, 100, 250, and 500 mL

7.17. Separatory funnels, typically 250 mL to 2-L size

7.18. Soxhlet/Dean-Stark (SDS) Extractor

7.19. Teflon boiling chips

7.20. Test tubes plus Teflon lined caps, 16 mm x 125 mm

7.21. Whatman GF/C, GF/D, and GF/F filters

8. REAGENTS, SOLVENTS AND STANDARDS

8.1. Reagents (Highest purity available)

8.1.1. Activated Silica Gel, for ~5 hours at 550^{o}C

8.1.2. Anhydrous sodium sulfate (Na_2SO_4) kilned for ~5 hours at 550^{o}C, granular

8.1.3. Hydromatrix

8.1.4. Ottawa sand, kilned for ~5 hours at 550^{o}C

8.1.5. Sodium hydroxide, 10N

8.1.6. Sulfuric acid, concentrated

8.1.7. Ultra-pure nitrogen gas

8.1.8. Water, HPLC

8.2. Solvents (Highest purity available)

8.2.1. Acetone

8.2.2. Ethanol

8.2.3. Hexane

8.2.4. Methanol

8.2.5. Methylene chloride (DCM)

8.2.6. Nonane (C_9)

8.2.7. Toluene

8.2.8. Tetradecane (C_{14})

8.3. Standards

8.3.1. All analytical standards are obtained from a certified vendor.

8.3.2. See current spike sheet for spiking concentrations and solutions.

8.4. See SOP 15 for preparation of reagents, standards and documentation.

9. SAMPLE COLLECTION, PRESERVATION, STORAGE AND HOLDING TIMES

9.1. Extract samples within 1 year and analyze within 1 year

9.2. Store samples at 4^{o}C (except fish/tissue at <-10^{o}C). Store extracts at <-10^{o}C in the dark.

9.3. If applicable, samples to be stored frozen are checked for adequate headspace prior to storage in case expansion occurs during freezing.

9.4. If residual chlorine is detected in an aqueous sample, add 80 mg sodium thiosulfate per liter.

9.5. Adjust to pH 2-3 with sulfuric acid (aqueous only).

10. SAMPLE PREPARATION

10.1. Residual Chlorine Determination (aqueous only)

10.1.1. Obtain an Aquacheck strip and place it directly into a small amount of sample in a disposable weigh boat. Move the strip back and forth for 30 seconds.

10.1.2. Check the color on the strip against the color chart on Aquacheck container.

10.1.3. If there is chlorine present, add 80 mg of sodium thiosulfate.

10.1.4. Record procedure on extraction benchsheet

10.2. pH Determination (aqueous only)

10.2.1. Obtain a pH strip and place it directly into a small amount of sample in a disposable weigh boat. Move the strip back and forth for 30 seconds.

10.2.2. Check the color on the strip against the color chart on the pH container. Adjust the pH to 2-3 with sulfuric acid if necessary.

10.3. Thawing Frozen Samples

10.3.1. Remove the sample from the freezer and place in a hood under ambient temperature.

- If there is a concern the sample container may break, place the container in a pre-cleaned secondary container.

10.4. Once the sample has thawed completely, proceed with sample preparation

10.5. Homogenization and Fillets

10.5.1. Remove any obviously extraneous materials and homogenize sample prior to sub-sampling for extraction. Homogenization may be as simple as shaking the container vigorously by hand to crushing, chopping, and use of a mechanical grinder. [redacted]

10.5.2. Fish Samples: Each whole fish filet is cut into dorsal/ventral strips ~2cm wide and shuffled prior to placing through a grinder to ensure proper homogenization unless the client requests otherwise. Once each fish filet is homogenized, the entire ground fish filet is mixed with the other designated ground fish filets to make a composite. The entire composite is placed in an amber glass jar for extraction. By client request, whole fish may be either homogenized or filleted prior to homogenization. All grinding parts and components are cleaned prior to homogenization and between each sample.

10.5.3. Wash with soap and water and rinse with [redacted]

10.5.4. [redacted] → [redacted] → [redacted] → [redacted]

10.6. % Solids Determination

10.6.1. "ZERO" or "TARE" the balance.

10.6.2. Place a weigh boat on the balance and record the weight as "Boat Weight".

10.6.3. Samples are individually homogenized with a clean spoon, Scoupula or spatula. Add a portion of the sample (2 – 10 g) to the weigh boat and record the weight as "Wet Wt. + Boat Wt."

10.6.4. Place the weigh boat plus sample in an oven at 110±5^{o}C for at least overnight.

10.6.5. Remove the weigh boat plus sample from the oven and allow to come to room temperature.

10.6.6. "ZERO" or "TARE" the balance.

10.6.7. Place the weigh boat plus sample on the balance and record the

weight as “Residue + Boat Wt.”

10.6.8. Calculate the percent solids by the following formula:

$$\%Solids = \frac{(Residue\ Wt. + Boat\ Wt.) - (Boat\ Wt.)}{(Wet\ Wt. + Boat\ Wt.) - (Boat\ Wt.)} X\ 100$$

10.6.9. For aqueous samples, if %Solid exceeds 1%, remove any rocks or stones and homogenize the sample prior to extraction. Weigh 10 grams dry-weight equivalent for extraction.

10.7. Compositing – by client request

10.7.1. Samples are individually homogenized, if necessary, with a clean spoon, Scoupula or spatula. Aqueous samples should be mixed and shaken to obtain a representative sample.

10.7.2. Weigh out approximately 50 grams, or amount designated by the client, from each individual sample and place into a pan.

10.7.3. Repeat the homogenization for each sample.

10.7.4. Place each individual sample into a new, separate container. Record the weight of each sample on the benchsheet.

10.7.5. Retain the original sample containers. The new container is given a new sample ID number and then processed through the appropriate extraction.

10.8. Sample Weight Determination

10.8.1. Volumetric: Allow sample to come to ambient temperature, mark the water meniscus on the side of the 1 L sample bottle. Once the sample has been transferred, fill the sample bottle to the mark with water and transfer to a 1000 mL graduated cylinder. Record the sample volume to the nearest 5 mL.

10.8.2. Gravimetric: Sample bottle including sample is placed on calibrated balance. The weight is recorded. The bottle is allowed to air-dry overnight and then re-weighed on a calibrated balance. This weight is recorded and percent solids are determined.

11. EXTRACTION PROCEDURES

11.1. Aqueous Samples Without Sediment/Particulates

11.1.1. Record the combined weight of the bottle, cap and sample for each sample to be extracted. After the sample has been removed from the bottle, allow it to drain overnight and reweigh it and the

cap to determine the amount of sample extracted.

11.1.2. For the method blank (MB) and OPR(s), transfer ~1 liter of HPLC water into a one liter bottle for each.

- Add the appropriate volume of Internal Standard (IS) solution to a test tube containing [redacted] of acetone. Quantitatively transfer to the aliquot of matrix with small portions of the solvent used. Add the appropriate volume of Native Standard (NS) solution to a test tube containing the IS/solvent and then quantitatively transfer to the aliquot of matrix assigned as an LCS, OPR, MS or MSD. Allow the spiked samples to equilibrate for at least 1 hour before extraction.

11.1.3. Pour the sample filtrate into a 2-liter separatory funnel. Rinse the sample filtrate container with [redacted] of DCM and add it to the separatory funnel.

11.1.4. Stopper each separatory funnel and shake vigorously, with frequent venting, for 2 minutes.

11.1.5. Allow the phases to separate (centrifugation or other mechanical means may be used to facilitate separation).

11.1.6. Drain the DCM extract through a funnel of pre-cleaned Na_2SO_4 into a 500 mL round bottom flask.

11.1.7. Extract the aqueous phase [redacted] portions of DCM ([redacted] pass the extracts through the Na_2SO_4 into a round bottom.

11.1.8. Spike the extract with the appropriate amount of CRS.

11.1.9. Concentrate the extract to approximately [redacted] continue with the cleanup.

11.2. Aqueous Samples Containing Sediment/Particulate

Option 1 – Default Procedure

11.2.1. Record the combined weight of the bottle, cap and sample for each sample to be extracted. After the sample has been removed from the bottle, allow it to drain overnight and reweigh it and the cap to determine the amount of sample extracted.

11.2.2. For the method blank (MB) and OPR(s), use [redacted] of sand for each.

- Add the appropriate volume of Internal Standard (IS) solution to a test tube containing [redacted] acetone. Quantitatively transfer to the aliquot of matrix with small portions of the

solvent used. Add the appropriate volume of Native Standard (NS) solution to a test tube containing the IS/solvent and then quantitatively transfer to the aliquot of matrix assigned as an LCS, OPR, MS or MSD. Allow the spiked samples to equilibrate for at least 1 hour before extraction.

11.2.3. Pre-clean [redacted] G/F-F and G/F-D filters using Buchner funnel and DCM.

11.2.4. Four solvent-rinse all parts of the HPF apparatus.

11.2.5. Assemble HPF apparatus using one each of DCM-cleaned [redacted] G/F-F and G/F-D filters.

11.2.6. Turn main nitrogen valve on with the flow rate set [redacted].

11.2.7. Pre-filter with [redacted] of HPLC water and discard (check canister for leaks).

11.2.8. Place a labeled beaker under HPF spout, slowly add sample to canister using a funnel. Rinse sample container once with [redacted] acetone, then 2 more rinses with HPLC water.

11.2.9. Remover beaker (save contents) and place sample container under spout.

11.2.10. Begin filtering, adjust filtering rate using valve on the HPF canister (main valve [redacted]

11.2.11. Add contents of beaker back to sample container with filtrate, replace beaker under spout, disassemble HPF apparatus, lift filters off the platform and collect any remaining water and combine with filtrate. Quickly transfer filters to soxhlet for toluene SDS extraction as in section 11.3

11.2.12. Pour filtrate into 2L separatory funnel and extract as in section 11.1

11.2.13. Combine aqueous and filter extracts and spike with CRS.

11.2.14. Continue with clean-up procedures.

Option 2

11.2.15. If the chemist determines by the appearance of the sample that HPF may not be an efficient procedure, Option 2 will be used.

11.2.16. Record the combined weight of the bottle, cap and sample for each sample to be extracted. After the sample has been removed

from the bottle, allow it to drain overnight and reweigh it and the cap to determine the amount of sample extracted.

11.2.17. For the method blank (MB) and OPR(s), use [redacted] of sand for each.

- Add the appropriate volume of Internal Standard (IS) solution to a test tube [redacted] of acetone. Quantitatively transfer to the aliquot of matrix with small portions of the solvent used. Add the appropriate volume of Native Standard (NS) solution to a test tube containing the IS/solvent and then quantitatively transfer to the aliquot of matrix assigned as an LCS, OPR, MS or MSD. Allow the spiked samples to equilibrate for at least 1 hour before extraction.

11.2.18. Pour entire sample into [redacted] centrifuge bottle. Rinse the sample container with HPLC water, and then rinse sample container with a small portion of DCM.

11.2.19. Centrifuge the samples [redacted] for [redacted] minutes.

11.2.20. Pour the aqueous portion into a separatory funnel and extract as in Section 11.1 above.

11.2.21. Transfer the sediment portion into [redacted] thimbles. Rinse the centrifuge container 2-3 times with toluene and transfer to thimble.

11.2.22. Extract sediment portion with toluene in SDS apparatus as in Section 11.3 below for 16-24 hrs.

11.2.23. Combine aqueous and sediment extracts, and spike with CRS

11.2.24. Continue with Clean-Up procedures.

➢ For DRBC samples, extract [redacted] of sample.

11.3. Soil, Sediment, Solids, Clay

11.3.1. Samples are individually homogenized with a clean spoon, scoupula or spatula. Weigh the sample (nominal 10 g dry weight equivalent) directly into [redacted] thimble, carefully breaking up any large lumps of sample.

- Add the appropriate volume of Internal Standard (IS) solution directly to the thimble. Add the appropriate volume of Native Standard (NS) to the thimbles assigned as an LCS, OPR, MS or MSD.

11.3.2. Reassemble the pre-extracted SDS apparatus, and add a fresh

charge of toluene to the receiver and reflux flask. Apply power to the heating mantle to begin refluxing. Adjust the reflux rate to match the rate of percolation until water removal lessens the restriction to toluene flow. [redacted]

11.3.3. Reflux the sample for a total of 16-24 hours (18-24 for tissues). Cool and disassemble the apparatus.

11.3.4. Remove the distilling flask. Drain the water from the Dean-Stark receiver.

11.3.5. Concentrate the extracts from the particles to approximately [redacted] using the rotary evaporator.

11.4. Sludge Samples

11.4.1. Weigh the sample (nominal [redacted] weight equivalent) directly into a kilned thimble.

- Place a ball [redacted] glass wool at the bottom of the thimble and the soxhlet.
- Add the appropriate volume of Internal Standard (IS) solution directly to the thimble. Add the appropriate volume of Native Standard (NS) to the thimbles assigned as an LCS, OPR, MS or MSD.

11.4.2. SDS extract for 16 – 24 hours with toluene.

11.4.3. If the % solids is too low to accommodate a [redacted] sample size into thimble, fill thimble half full of Hydromatrix.

11.4.4. Tare weight of thimble then fill thimble full of sludge.

11.4.5. Record the weight of sludge, spike and then transfer immediately to soxhlet.

11.5. Tissue

11.5.1. Mix 10 g of well ground fish/tissue with at least [redacted] of pre-cleaned Na_2SO_4 in a beaker. (If 25 g of tissue are used, then [redacted] of Na_2SO_4 is used.)

11.5.2. Stir frequently to remove any lumps.

11.5.3. Re-stir after about [redacted] and transfer the mixture to a [redacted] thimble.

- Add the appropriate volume of Internal Standard (IS) solution directly to the thimble. Add the appropriate volume of Native

Standard (NS) to the thimbles assigned as an LCS, OPR, MS or MSD.

11.5.4. Soxhlet extract for 18-24 hours with [redacted] DCM:Hexane

11.5.5. Add the appropriate amount of Cleanup Recovery Standard(s) (CRS) to the extract.

11.5.6. Concentrate the extract to [redacted]

11.5.7. If % Lipids are to be determined proceed to the next step. Otherwise proceed with the appropriate cleanup procedures.

11.6. % Lipids

11.6.1. Transfer to a 250 mL mixing cylinder, adjust the extract to a [redacted] volume [redacted] using [redacted] DCM:Hexane and mix well.

11.6.2. Transfer 10% of the solution to an aluminum dish that has been pre-weighed on an analytical balance.

11.6.3. Allow the extract to air dry completely and then place in a 50±5 °C oven overnight.

11.6.4. When the aliquot is dry, allow to cool to room temperature, re-weigh the dish on an analytical balance and record the weight. Calculate the % lipids using the following equation:

$$\%\,\text{lipids} = \frac{\text{lipid residue weight}}{10\%\ \text{of sample weight}} \text{x}100$$

11.6.5. Using the remaining 90% of the extract, proceed with the appropriate cleanup procedures.

12. CLEANUP PROCEDURES

12.1. Coplanar PCBs analyses

12.1.1. Exchange the extract solvent to hexane and concentrate by one of the following techniques.

- Using a rotary evaporator, add [redacted] C_{14}; rotovap to the C_{14}, add [redacted] of hexane and rotovap to the C_{14}.
- Using a turbovap evaporator; evaporate to [redacted], add [redacted] of hexane, be sure to mix the solvent in the stem with the added hexane and evaporate to [redacted].

12.1.2. Proceed to the appropriate cleanup.

12.2. Total PCBs analyses

12.2.1. [redacted]

12.2.2. Rotovap to [redacted], add [redacted] hexane and Rotovap to [redacted] repeat once more. Do not let go to dryness.

12.2.3. Proceed to the appropriate cleanup.

12.3. Acid Partitioning (AP)

12.3.1. Rotovap to [redacted] Add sample to pre-cleaned separatory funnel. Add 80 mL hexane.

12.3.2. Carefully add ~50 mL of concentrated H_2SO_4 to a separatory funnel.

12.3.3. Shake for approximately 30 seconds with periodic venting, allow the layers to separate (centrifugation may be necessary) and discard the acid layer. Add ~50 mL of HPLC water to the separatory funnel. Shake for approximately 30 seconds with venting and discard the aqueous layer. Repeat if sample still contains color.

12.3.4. Pass the organic layer through Na_2SO_4 (pre-rinsed with 2 ~15 mL aliquots of hexane) then concentrate to <10 mL.

12.4. Acid Base Silica Gel (ABSG)

12.4.1. Prepare the column as depicted in Figure 1.

12.4.2. All traces of solvents other than hexane must be removed from the extract.

12.4.3. Adjust extract volume to <10 mL.

12.4.4. Rinse the ABSG column with [redacted] hexane, discard the eluate.

12.4.5. Transfer the extract to the column with 2-4 small portions of hexane, begin collecting the eluate.

12.4.6. When the extract reaches the sodium sulfate, add [redacted] of hexane.

12.4.7. Concentrate the eluate appropriately.

12.5. Acid Alumina (AA) - Coplanar PCBs only.

12.5.1. Prepare column as depicted in Figure 2

12.5.2. Rinse column with [redacted] DCM and then [redacted] of hexane, discard the eluate.

12.5.3. Adjust extract volume to [redacted].

12.5.4. Transfer the extract to the column with [redacted] portions of hexane. Discard the eluate.

12.5.5. Elute the column with [redacted] hexane. Discard the eluate.

12.5.6. Elute column with [redacted] DCM:Hexane, collect the eluate.

12.5.7. Concentrate the eluate to [redacted]

13. ADJUST TO FINAL VOLUME

13.1. Coplanar PCBs (Solid)

13.1.1. Using hexane, quantitatively transfer the concentrated eluate to a conical vial that contains the Recovery Standard (RS) and 40 L of tetradecane.

13.1.2. Using nitrogen blow down, concentrate to the tetradecane.

13.1.3. Rinse the walls of the conical vial with hexane, re-blow down to the tetradecane.

13.1.4. Using a 50-100 L Wiretrol, transfer the tetradecane to an insert in a crimp top autoinjector vial and then cap.

13.2. Coplanar PCBs (Aqueous and Tissue)

13.2.1. Using hexane, quantitatively transfer the concentrated eluate to a conical vial that contains the Recovery Standard (RS) and [redacted] of tetradecane.

13.2.2. Using nitrogen blow down, concentrate to the tetradecane.

13.2.3. Rinse the walls of the conical vial with hexane, re-blow down to the tetradecane.

13.2.4. [redacted], transfer the tetradecane to an insert in a crimp top autoinjector vial and then cap.

13.3. Total PCBs

13.3.1. Aqueous

- Add [redacted] to a conical vial and quantitatively transfer sample to conical vial with hexane, blow down to [redacted] transfer the concentrate to an amber crimp top autoinjector vial, with an insert, and then cap.

13.3.2. All other matrices

- Add [redacted] of C_9 to a conical vial and quantitatively transfer sample to conical vial with hexane, blow down to [redacted] and add appropriate amount of RS. Using nitrogen blow down, [redacted] concentrate to C_9. [redacted], transfer the concentrate to an amber crimp top autoinjector vial, with an insert, and then cap.

14. QUALITY CONTROL

14.1. Method Blank (MB): Method Blank is a matrix preparation that is free from native analyte that has been prepared and analyzed using the same procedures followed for the rest of the sample batch. HPLC water is used for aqueous samples, sand is used for solid and fish samples. See Table 9 for more information.

14.2. Ongoing Precision and Recovery (OPR): An ongoing precision and recovery sample is prepared by adding a known quantity of native standard to an interferent free matrix and used to assess method performance (precision and recovery). See Table 9 for more information.

14.2.1. Spike 10 L of compound spiking solution into the sample.

14.3. Matrix Spike (MS/MSD): A matrix spike sample is prepared by adding a known quantity of native standard to a sample matrix prior to extraction. See Table 9 for more information.

Note: Projects performed pursuant to the guidelines established by the DOD QSM shall contain an associated Matrix Spike per preparatory batch. A Matrix Spike Duplicate or Laboratory Duplicate shall also be analyzed per preparatory batch for these projects. See Table 9 for more information.

14.3.1. Spike 10 L of compound spiking solution into the sample.

14.4. Duplicate Samples: Duplicate samples are two separate aliquots taken from the same source. Duplicate samples are analyzed independently to assess laboratory precision. Duplicate samples are performed by client request. See Table 9 for more information.

15. HRMS ANALYSIS

15.1. Establish the necessary operating conditions. The GC conditions may be optimized for compound separation and sensitivity. Once optimized, the same GC conditions must be used for the analysis of all standards, blanks, OPR aliquots and samples. The following GC operating conditions are for guidance and adjustments may be required.

Injector temperature: [redacted]
Interface temperature: [redacted]
Initial temperature: [redacted]
Initial time: [redacted]
Temperature program: [redacted] C at [redacted]
[redacted]
[redacted]
[redacted]
[redacted]
[redacted]

15.2. GC Column

15.2.1. The retention time for decachlorobiphenyl must be greater than 55 minutes, for a ZB-1 column and SPB-Octyl column or equivalent.

15.2.2. The ZB -1 column must resolve the following congeners:

- 66, 70, 74, 80 with a valley height less than 40%
- 123, 118 with a valley height less than 40%
- 156, 157 with a valley height less than 40%

15.2.3. The SPB-Octyl column must resolve the following congeners:

- 34, 23 with a valley height less than 40%
- 187, 182 with a valley height less than 40%
- 156, 157 must co-elute within 2 seconds of peak maximum

15.2.4. If a project/client requires separation of any co-eluting PCB congeners, then the extract may be analyzed on an alternate GC column.

15.3. Instrument Tuning

15.3.1. Inject the reference compound perfluorokerosene (PFK) performed at the beginning and at the end of each shift. PFK provides the required lock masses and is used for tuning the

mass spectrometer.

15.3.2. Using a PFK molecular leak, tune the instrument to meet the minimum required resolving power of 10,000 at or close to m/z 304.9824. For each descriptor, monitor and record the resolution and exact m/z of three to five reference peaks covering the range of the descriptor. See Table 9 for more information.

15.4. Initial Calibration

15.4.1. Under the same conditions, inject 1-2 L of each of the six calibration solutions containing all PCB isomers. Calibration standard solutions are presented in Table 2. For consistency, all samples and standards will have the same injection volume.

15.4.2. Forty-four internal standards and six recovery standards are used to improve quantitation.

See Table 9 for more information and corrective action.

15.5. Set up the analytical run following this sequential injection pattern: Window Defining Mix (CS3), OPR, Solvent Blank, Method Blank, Samples, Solvent Blank, and CS3 (if required).

15.6. Continuing Calibration

15.6.1. Inject a mid-range standard from the initial calibration curve (CS3). The following criteria must be met:

1.) Calculate the concentration of each native compound either by isotope dilution or internal standard technique. Each compound must be within the verification limits established in Table 7.

2.) The ion ratios must be within the theoretical ratio limits (Table 3).

3.) The signal to noise ratio (s/n) must exceed 10:1 for all ions monitored.

4.) The absolute retention times of the internal standards shall be within ±15 seconds of the retention times obtained during calibration.

5.) The relative retention times of the peak for a native and labeled PCB should be within 0.5% of the retention time windows established from the initial calibration curve.

15.7. Qualitative Determination

15.7.1. A chromatographic peak is identified as a PCB or labeled compound when all of the following criteria are met:

1.) The signals for the two exact m/z's being monitored (Table 8 of Method 1668) must be present and must maximize within ±2 seconds of one another.

2.) The signal-to-noise ratio (S/N) of each of the two exact m/z's must be ≥2.5:1 for a sample extract, and ≥10:1 for a calibration standard.

3.) The ratio of the integrated areas of the two exact m/z's must be within the limits established in Table 3.

- Optional reporting: If the ion abundance ratio is outside the limits established in Table 3, the chromatographic peak will be quantified as an estimated maximum possible concentration or EMPC.

4.) The relative retention times of the peak for a native and labeled PCB should be within 0.5% of the retention time windows established from the initial calibration curve.

- If the above mentioned criteria are not met, the peak is not identified as a positive.

15.8. Quantitative Determination

15.8.1. Any numerical values that are calculated below the quantitation limit are reported as non-detects unless requested otherwise by client. Coplanar PCBs are reported as non-detects below the detection limit and may be qualified with a "J" flag if detected below the quantitation limit.

15.8.2. For peaks which meet the criteria listed above, quantitate the PCB peaks from the response relative to the appropriate internal standard.

15.8.3. Any peaks representing the other congeners are quantitated using an average of the response factors from all of the labeled PCBs isomers at the same level of chlorination.

15.8.4. Recovery of each internal standard must be within limits in Table 7 for samples.

15.8.5. It the above-mentioned criteria are not met but the qualitative criteria is met, then the data are qualified appropriately.

- For DRBC samples, analyze on SPB-Octyl column.

16. CALCULATIONS

16.1. The concentrations for PCB compounds are calculated by using the formula:

$$C_X = \frac{(A_X)(Q_{IS})}{(A_{IS})(W)(RRF)}$$

Where:

C_X	=	*Concentration of unlabeled PCB congeners (or group of coeluting isomers within an homologous series),*
A_X	=	*Sum of the integrated ion abundances of the quantitation ions for unlabeled PCBs*
A_{IS}	=	*Sum of the integrated ion abundances of the quantitation ion for the labeled internal standards,*
Q_{IS}	=	*Quantity, in pg, of the internal standard added to the sample before extraction,*
W	=	*Weight of the sample (solid, dry weight or liquid)*
DW	=	*Sample wt.* $\times$ *%solids/100*
RRF	=	*Calculated relative response factor for the analyte.*

16.2. The detection limits can be calculated using the following formula:

$$DL = \frac{(2.5)(H_N)(Q_{IS})}{(H_{IS})(W)(RRF)}$$

Where:

DL	=	*Sample specific estimated detection limit,*
H_N	=	*Noise height (peak to peak),*
H_{IS}	=	*Peak height of the internal standard,*
Q_{is}	=	*Quantity, in pg, of the internal standard added to the sample before extraction,*
W	=	*Weight of the sample (solid or liquid), and*
RRF	=	*Calculated relative response factor for the analyte.*

16.3. The reporting limits can be calculated using the following formula:

$$RL = \frac{(\text{Extract Conc. of Low point of curve})(\text{final volume})}{(\text{Weight of sample})} \rightarrow (\text{Split})$$

16.4. The Relative Response factor can be calculated using the following formula:

$$RRF = \frac{\left(A^1_N + A^2_N\right)(C_{IS})}{\left(A^1_{IS} + A^2_{IS}\right)(C_N)}$$

Where:

$A1_N$, $A2_N$ = *Areas of the primary and secondary m/z's for the native compound*

$A1_{IS}$, $A2_{IS}$ = *Areas of the primary and secondary m/z's for the labeled compound.*

C_{IS} = *Concentration of the labeled compound in the calibration standard.*

C_N = *Concentration of the native compound in the calibration standard*

16.5. Estimated Maximum Possible Concentration (EMPC):

$$EMPC = \frac{(A_X)\,(Q_{IS})}{(A_{IS})\,(W)\,(RF_N)}$$

Where:

A_X = *Sum of the area of the smaller peak and of the other peak area calculated using the theoretical chlorine isotope ratio*

A_{IS} = *Sum of the integrated ion abundances of the quantitation ions for the labeled internal standards*

Q_{IS} = *Quantity, in pg, of the internal standard added to the sample before extraction*

W = *Weight of the sample or volume of aqueous sample*

RF_N = *Calculated mean relative response factor for the analyte*

17. POLLUTION PREVENTION

17.1. The solvent evaporation techniques used in this method are amenable to solvent recovery, and it is recommended that the laboratory recover solvents wherever feasible.

17.2. Standards should be prepared in volumes consistent with laboratory use to minimize disposal of standards.

18. WASTE MANAGEMENT

18.1. Waste generated in the procedure must be segregated and disposed according to the facility hazardous waste procedures. Safety officer should be contacted if additional information is required.

18.2. The laboratory waste management is in compliance with all federal, state, and local regulations to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations.

19. METHOD PERFORMANCE

19.1. This SOP is based on the following method: EPA Method 1668, Revision A: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, and Tissue by HRGC/HRMS, United States Office of Water, EPA No. EPA 821-R-00-002, Environmental Protection Agency (4303), December 1999

20. REFERENCES

20.1. Alford-Stevens, A., Bellar, T. A., Eichelberger, J. W., and W. L. Budde, 1984. Method 680 -- Determination of Pesticides and PCBs in Water and Soil/Sediment by HRGC/LRMS, U. S. EPA. Cincinnati, OH.

20.2. Draft Method 1668. Measurement of Toxic PCB Congeners By Isotope Dilution HRGC/HRMS, Prepared by Analytical Methods Staff, Engineering and Analysis Division (4303), Office of Science and Technology, Office of Water, U. S. Environmental Protection Agency, Washington, DC, March, 1997.

20.3. EPA Region 10 SOP For the Validation of Method 1668 Toxic, Dioxin-Like, PCB Data. Revision 1.0, December 8, 1995.

20.4. Method 1668, Revision A: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, and Tissue by HRGC/HRMS. Prepared under the direction of William A. Telliard, Engineering and Analysis Division, Office of Water, U.S. Environmental Protection Agency, Washington D.C., August 2003

20.5. Vista Analytical Laboratory SOP 9, Manual Integrations

20.6. SOP 10 - Instrument Maintenance Logbooks and Schedule

20.7. Method 1668C: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, and Tissue by HRGC/HRMS. U.S. Environmental Protection Agency, Office of Water, Office of Science and Technology, Engineering and Analysis Division (4303T), Washington D.C., April 2010.

Figure 1

Acid Base Silica Gel (ABSG)

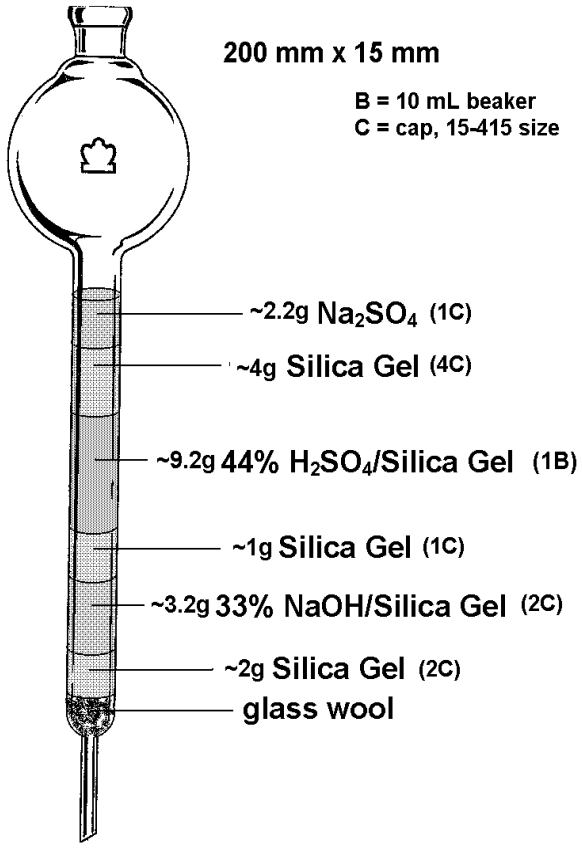


Figure 2

Acid Alumina (AA) Coplanar PCBs only

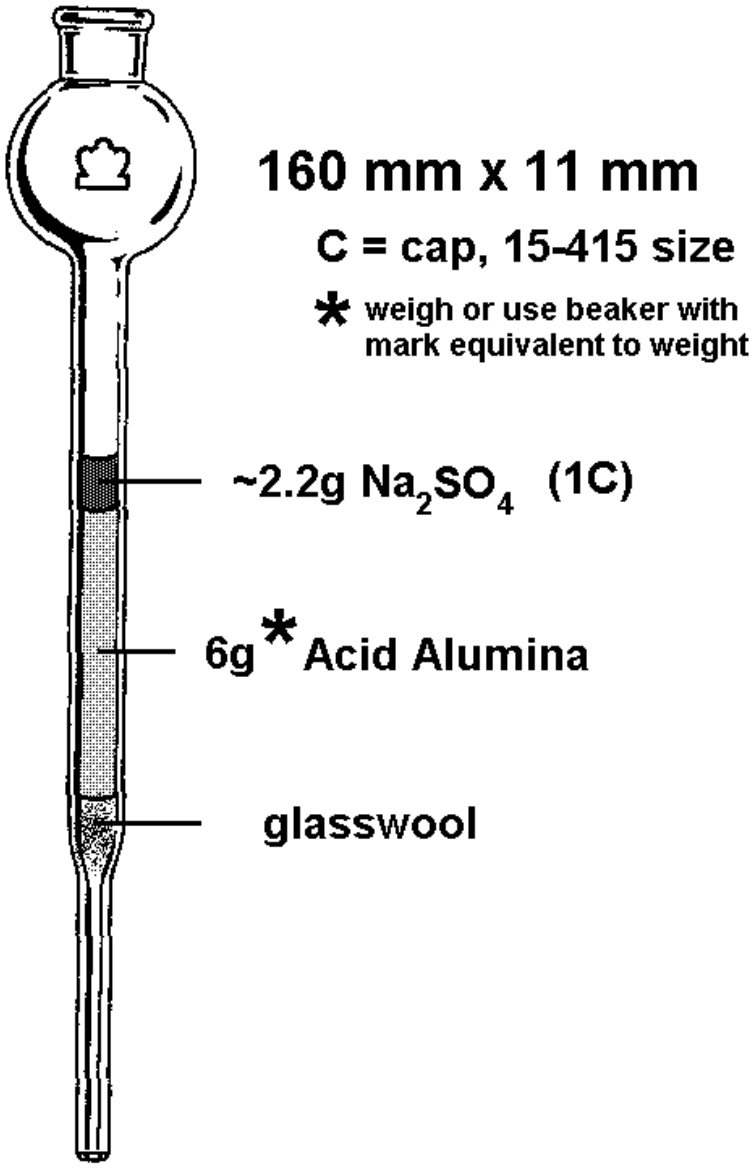


Table 1

PCB Target Compounds and Quantitation Limits

Coplanar PCB Congeners	Congener No.	Water (pg/L)	Solid (pg/g)	Fish/Tissue (pg/g)	Serum (pg/g)	Serum (pg/g-Lipid)
3,3',4,4'-Tetra-CB	77	5.0	2.5	0.5	0.25	50.0
3,4,4',5-Tetra-CB	81	5.0	2.5	0.5	0.25	50.0
3,3',4,4',5-Penta-CB	126	5.0	2.5	0.5	0.25	50.0
3,3',4,4',5,5'-Hexa-CB	169	5.0	2.5	0.5	0.25	50.0
Toxically Significant Mono-Ortho Substituted PCBs						
2,3,3',4,4'-Penta-CB	105	5.0	2.5	0.5	0.25	50.0
2,3,4,4',5-Penta-CB	114	5.0	2.5	0.5	0.25	50.0
2,3',4,4',5-Penta-CB	118	5.0	2.5	0.5	0.25	50.0
2',3,4,4',5-Penta-CB	123	5.0	2.5	0.5	0.25	50.0
2,3,3',4,4',5-Hexa-CB	156	5.0	2.5	0.5	0.25	50.0
2,3,3',4,4',5'-Hexa-CB	157	5.0	2.5	0.5	0.25	50.0
2,3',4,4',5,5'-Hexa-CB	167	5.0	2.5	0.5	0.25	50.0
2,3,3',4,4',5,5'-Hepta-CB	189	5.0	2.5	0.5	0.25	50.0
Other Environmentally Significant PCBs (209)						
2-Mono-CB	1	5.0	2.5	0.5	0.25	50.0
3-Mono-CB	2	5.0	2.5	0.5	0.25	50.0
4-Mono-CB	3	5.0	2.5	0.5	0.25	50.0
2,2'-Di-CB 2,6-Di-CB	4/10	10.0	10.0	1.0	0.5	100.0
2,3'-Di-CB	6	5.0	2.5	0.5	0.25	50.0
2,3-Di-CB 2,4'-Di-CB	5/8	10.0	5.0	1.0	0.50	100.0
2,4-Di-CB 2,5-Di-CB	7/9	10.0	5.0	1.0	0.50	100.0
3,3'-Di-CB	11	5.0	2.5	0.5	0.25	50.0
3,4-Di-CB 3,4'-Di-CB	12/13	10.0	5.0	1.0	0.50	100.0
3,5-Di-CB	14	5.0	2.5	0.5	0.25	50.0
4,4'-Di-CB	15	5.0	2.5	0.5	0.50	50.0
2,2',3-Tri-CB 2,4',6-TrCB	16/32	10.0	5.0	1.0	0.50	100.0
2,2',4-Tri-CB	17	5.0	2.5	0.5	0.25	50.0
2,2',5-Tri-CB	18	5.0	2.5	0.5	0.25	50.0
2,2',6-Tri-CB	19	5.0	2.5	0.5	0.25	50.0
2,3,3'-Tri-CB 2,3,4-Tri-CB 2',3,4-Tri-CB	20/21/33	15.0	7.5	1.5	0.75	150.0
2,3,4'-Tri-CB	22	5.0	2.5	0.5	0.25	50.0

Table 1

PCB Target Compounds and Quantitation Limits

PCB Congeners	Congener No.	Water (pg/L)	Solid (pg/g)	Fish/Tissue (pg/g)	Serum (pg/g)	Serum (pg/g-Lipid)
2,3,5-Tri-CB	23	5.0	2.5	0.5	0.25	50.0
2,3,6-Tri-CB 2,3',6-Tri-CB	24/27	10.0	5.0	1.0	0.50	100.0
2,3',4-Tri-CB	25	5.0	2.5	0.5	0.25	50.0
2,3',5-Tri-CB	26	5.0	2.5	0.5	0.25	50.0
2,4,4'-Tri-CB	28	5.0	2.5	0.5	0.25	50.0
2,4,5-Tri-CB	29	5.0	2.5	0.5	0.25	50.0
2,4,6-Tri-CB	30	5.0	2.5	0.5	0.25	50.0
2,4',5-Tri-CB	31	5.0	2.5	0.5	0.25	50.0
2'3,5-Tri-CB	34	5.0	2.5	0.5	0.25	50.0
3,3',4-Tri-CB	35	5.0	2.5	0.5	0.25	50.0
3,3',5-Tri-CB	36	5.0	2.5	0.5	0.25.	50.0
3,4,4'-Tri-CB	37	5.0	2.5	0.5	0.25	50.0
3,4,5-Tri-CB	38	5.0	2.5	0.5	0.25	50.0
3,4',5-Tri-CB	39	5.0	2.5	0.5	0.25	50.0
2,2',3,3'-Tetra-CB	40	5.0	2.5	0.5	0.25	50.0
2,2',3,4-Tetra-CB 2,3,4',6-Tetra-CB 2,3',4',6-Tetra-CB 2,3',5,5'-Tetra-CB	41/64/71/72	20.0	10.0	2.0	1.00	200.0
2,2',3,4'-Tetra-CB 2,3,3',6-Tetra-CB	42/59	10.0	5.0	1.0	0.50	100.0
2,2',3,5-Tetra-CB 2,2',4,5'-Tetra-CB	43/49	10.0	5.0	1.0	0.50	100.0
2,2',3,5'-Tetra-CB	44	5.0	2.5	0.5	0.25	50.0
2,2',3,6-Tetra-CB	45	5.0	2.5	0.5	0.25	50.0
2,2',3,6'-Tetra-CB	46	5.0	2.5	0.5	0.25	50.0
2,2',4,4'-Tetra-CB	47	5.0	2.5	0.5	0.25	50.0
2,2',4,5-Tetra-CB 2,4,4',6-Tetra-CB	48/75	10.0	5.0	1.0	0.50	100.0
2,2',4,6-Tetra-CB	50	5.0	2.5	0.5	0.25	50.0
2,2',4,6'-Tetra-CB	51	5.0	2.5	0.5	0.25	50.0
2,2',5,5'-Tetra-CB 2,3',4,6-Tetra-CB	52/69	10.0	5.0	1.0	0.50	100.0
2,2',5,6'-Tetra-CB	53	5.0	2.5	0.5	0.25	50.0
2,2',6,6'-Tetra-CB	54	5.0	2.5	0.5	0.25	50.0
2,3,3',4-Tetra-CB	55	5.0	2.5	0.5	0.25	50.0
2,3,3',4'-Tetra-CB 2,3,4,4'-Tetra-CB	56/60	10.0	5.0	1.0	0.50	100.0

Table 1

PCB Target Compounds and Quantitation Limits

PCB Congeners	Congener No.	Water (pg/L)	Solid (pg/g)	Fish/Tissue (pg/g)	Serum (pg/g)	Serum (pg/g-Lipid)
2,3,3',5-Tetra-CB	57	5.0	2.5	0.5	0.25	50.0
2,3,3',5'-Tetra-CB	58	5.0	2.5	0.5	0.25	50.0
2,3,4,5-Tetra-CB 2,3',4',5-Tetra-CB	61/70	10.0	5.0	1.0	0.50	100.0
2,3,4,6-Tetra-CB	62	5.0	2.5	0.5	0.25	50.0
2,3,4',5-Tetra-CB	63	5.0	2.5	0.5	0.25	50.0
2,3,5,6-Tetra-CB	65	5.0	2.5	0.5	0.25	50.0
2,3',4,5-Tetra-CB	67	5.0	2.5	0.5	0.25	50.0
2,3',4,5'-Tetra-CB	68	5.0	2.5	0.5	0.25	50.0
2,3',5',6-Tetra-CB	73	5.0	2.5	0.5	0.25	50.0
2,4,4',5-Tetra-CB	74	5.0	2.5	0.5	0.25	50.0
2',3,4,5-Tetra-CB 2,3',4,4'-Tetra-CB	76/66	10.0	5.0	1.0	0.50	100.0
3,3',4,4'-Tetra-CB	77	5.0	2.5	0.5	0.25	50.0
3,3',4,5-Tetra-CB	78	5.0	2.5	0.5	0.25	50.0
3,3',4,5'-Tetra-CB	79	5.0	2.5	0.5	0.25	50.0
3,3',5,5'-Tetra-CB	80	5.0	2.5	0.5	0.25	50.0
3,4,4',5-Tetra-CB	81	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4-Penta-CB	82	5.0	2.5	0.5	0.25	50.0
2,2',3,3',5-Penta-CB	83	5.0	2.5	0.5	0.25	50.0
2,2',3,3',6-Penta-CB 2,2',3,5,5'-Penta-CB	84/92	10.0	5.0	1.0	0.50	100.0
2,2',3,4,4'-Penta-CB 2,3,4,5,6-Penta-CB	85/116	10.0	5.0	1.0	0.50	100.0
2,2',3,4,5-Penta-CB	86	5.0	2.5	0.5	0.25	50.0
2,2',3,4,5'-Penta-CB 2,3,4',5,6-Penta-CB 2',3,4,5,6'-Penta-CB	87/117/125	15.0	7.5	1.5	0.75	150.0
2,2',3,4,6-Penta-CB 2,2',3,4',6-Penta-CB	88/91	10.0	5.0	1.0	0.50	100.0
2,2',3,4,6'-Penta-CB	89	5.0	2.5	0.5	0.25	50.0
2,2',3,4',5-Penta-CB 2,2',4,5,5'-Penta-CB	90/101	10.0	5.0	1.0	0.50	100.0
2,2',3,5,6-Penta-CB	93	5.0	2.5	0.5	0.25	50.0
2,2',3,5,6'-Penta-CB	94	5.0	2.5	0.5	0.25	50.0
2,2',3,5',6-Penta-CB 2,2',3',4,6-Penta-CB 2,2',4,5,6'-Penta-CB	95/98/102	15.0	7.5	1.5	0.75	150.0

Table 1

PCB Target Compounds and Quantitation Limits

PCB Congeners	Congener No.	Water (pg/L)	Solid (pg/g)	Fish/Tissue (pg/g)	Serum (pg/g)	Serum (pg/g-Lipid)
2,2',3,6,6'-Penta-CB	96	5.0	2.5	0.5	0.25	50.0
2,2',3,4',5-Penta-CB	97	5.0	2.5	0.5	0.25	50.0
2,2',4,4',5-Penta-CB	99	5.0	2.5	0.5	0.25	50.0
2,2',4,4',6-Penta-CB	100	5.0	2.5	0.5	0.25	50.0
2,2',4,5',6-Penta-CB	103	5.0	2.5	0.5	0.25	50.0
2,2',4,4,6'-Penta-CB	104	5.0	2.5	0.5	0.25	50.0
2,3,3',4,4'-Penta-CB	105	5.0	2.5	0.5	0.25	50.0
2,3',4,4',5-Penta-CB 2,3,3',4,5-Penta-CB	118/106	10.0	5.0	1.0	0.50	100.0
2,3,3',4',5-Penta-CB 2,3,3',4,6-Penta-CB	107/109	10.0	5.0	1.0	0.50	100.0
2,3,3',4,5'-Penta-CB 2,3,3',5,6-Penta-CB	108/112	10.0	5.0	1.0	0.50	100.0
2,3,3',4',6-Penta-CB	110	5.0	2.5	0.5	0.25	50.0
2,3,3',5,5'-Penta-CB 2,3,4,4',6-Penta-CB	111/115	10.0	5.0	1.0	0.50	100.0
2,3,3',5',6-Penta-CB	113	5.0	2.5	0.5	0.25	50.0
2,3,4,4',5-Penta-CB	114	5.0	2.5	0.5	0.25	50.0
2,3',4,4',6-Penta-CB	119	5.0	2.5	0.5	0.25	50.0
2,3',4,5,5'-Penta-CB	120	5.0	2.5	0.5	0.25	50.0
2,3',4,5',6-Penta-CB	121	5.0	2.5	0.5	0.25	50.0
2'3,3'4,5-PentaCB	122	5.0	2.5	0.5	0.25	50.0
2',3,4,4'5-Penta-CB	123	5.0	2.5	0.5	0.25	50.0
2',3,4,5,5'-Penta-CB	124	5.0	2.5	0.5	0.25	50.0
3,3'4,4',5-Penta-CB	126	5.0	2.5	0.5	0.25	50.0
3,3',4,5,5'-Penta-CB	127	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,4'-Hexa-CB 2,3,3',4',5,5'-Hexa-CB	128/162	10.0	5.0	1.0	0.50	100.0
2,2',3,3',4,5-Hexa-CB	129	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,5'-Hexa-CB	130	5.0	2.5	0.5	0.25	50.0
2,2'3,3'4,6-Hexa-CB 2,2',3,3'5,5'-Hexa-CB	131/133	10.0	5.0	1.0	0.50	100.0
2,2',3,3',4,6'-Hexa-CB 2,3,3',4,5',6-Hexa-CB	132/161	10.0	5.0	1.0	0.50	100.0
2,2',3,3',5,6-Hexa-CB 2,2',3,4,5,6'-Hexa-CB	134/143	10.0	5.0	1.0	0.50	100.0
2,2',3,3',5,6'-Hexa-CB	135	5.0	2.5	0.5	0.25	50.0
2,2',3,3',6,6'-Hexa-CB	136	5.0	2.5	0.5	0.25	50.0
2,2',3,4,4',5-Hexa-CB	137	5.0	2.5	0.5	0.25	50.0

Table 1

PCB Target Compounds and Quantitation Limits

PCB Congeners	Congener No.	Water (pg/L)	Solid (pg/g)	Fish/Tissue (pg/g)	Serum (pg/g)	Serum (pg/g-Lipid)
2,2',3,4,4',5'-Hexa-CB 2,3,3',4',5,6-Hexa-CB 2,3,3',4',5',6-Hexa-CB	138/163/164	15.0	7.5	1.5	0.75	150.0
2,2',3,4,4',6-Hexa-CB 2,2',3,4',5',6-Hexa-CB	139/149	10.0	5.0	1.0	0.50	100.0
2,2',3,4,4',6'-Hexa-CB	140	5.0	2.5	0.5	0.25	50.0
2,2',3,4,5,5'-Hexa-CB	141	5.0	2.5	0.5	0.25	50.0
2,2',3,4,5,6-Hexa-CB	142	5.0	2.5	0.5	0.25	50.0
2,2',3,4,5',6-Hexa-CB	144	5.0	2.5	0.5	0.25	50.0
2,2',3,4,6,6'-Hexa-CB	145	5.0	2.5	0.5	0.25	50.0
2,2',3,4',5,5'-Hexa-CB 2,3,3',5,5',6-Hexa-CB	146/165	10.0	5.0	1.0	0.50	100.0
2,2',3,4',5,6-Hexa-CB	147	5.0	2.5	0.5	0.25	50.0
2,2',3,4',5,6'-Hexa-CB	148	5.0	2.5	0.5	0.25	50.0
2,2',3,4',6,6'-Hexa-CB	150	5.0	2.5	0.5	0.25	50.0
2,2',3,5,5',6-Hexa-CB	151	5.0	2.5	0.5	0.25	50.0
2,2',3,5,6,6'-Hexa-CB	152	5.0	2.5	0.5	.0.25	50.0
2,2',4,4',5,5'-Hexa-CB	153	5.0	2.5	0.5	0.25	50.0
2,2'4,4',5',6-Hexa-CB	154	5.0	2.5	0.5	0.25	50.0
2,2',4,4',6,6'-Hexa-CB	155	5.0	2.5	0.5	0.25	50.0
2,3,3',4,4',5-Hexa-CB	156	5.0	2.5	0.5	0.25	50.0
2,3,3',4,4',5'-Hexa-CB	157	5.0	2.5	0.5	0.25	50.0
2,3,3',4,4',6-Hexa-CB 2,3,3',4,5,6-Hexa-CB	158/160	10.0	5.0	1.0	0.50	100.0
2,3,3',4,5,5'-Hexa-CB	159	5.0	2.5	0.5	0.25	50.0
2,3,4,4',5,6-Hexa-CB	166	5.0	2.5	0.5	0.25	50.0
2,3',4,4',5,5'-Hexa-CB	167	5.0	2.5	0.5	0.25	50.0
2,3',4,4',5',6-Hexa-CB	168	5.0	2.5	0.5	0.25	50.0
3,3'4,4',5,5'-Hexa-CB	169	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,4',5-Hepta-CB	170	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,4',6-Hepta-CB	171	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,5,5'-Hepta-CB	172	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,5,6-Hepta-CB	173	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,5,6'-Hepta-CB	174	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,5',6-Hepta-CB	175	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,6,6'-Hepta-CB	176	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4',5,6-Hepta-CB	177	5.0	2.5	0.5	0.25	50.0
2,2',3,3',5,5',6-Hepta-CB	178	5.0	2.5	0.5	0.25	50.0
2,2',3,3',5,6,6'-Hepta-CB	179	5.0	2.5	0.5	0.25	50.0
2,2',3,4,4',5,5'-Hepta-CB	180	5.0	2.5	0.5	0.25	50.0

Table 1

PCB Target Compounds and Quantitation Limits

2,2',3,4,4',5,6-Hepta-CB	181	5.0	2.5	0.5	0.25	50.0
PCB Congeners	**Congener No.**	**Water (pg/L)**	**Solid (pg/g)**	**Fish/Tissue (pg/g)**	**Serum (pg/g)**	**Serum (pg/g-Lipid)**
2,2',3,4,4',5,6'-Hepta-CB 2,2',3,4',5,5',6-Hepta-CB	182/187	10.0	5.0	1.0	0.50	100.0
2,2',3,4,4',5',6-Hepta-CB	183	5.0	2.5	0.5	0.25	50.0
2,2',3,4,4',6,6'-Hepta-CB	184	5.0	2.5	0.5	0.25	50.0
2,2',3,4,5,5',6-Hepta-CB	185	5.0	2.5	0.5	0.25	50.0
2,2',3,4,5,6,6'-Hepta-CB	186	5.0	2.5	0.5	0.25	50.0
2,2',3,4',5,6,6'-Hepta-CB	188	5.0	2.5	0.5	0.25	50.0
2,3,3',4,4',5,5'-Hepta-CB	189	5.0	2.5	0.5	0.25	50.0
2,3,3',4,4',5,6-Hepta-CB	190	5.0	2.5	0.5	0.25	50.0
2,3,3',4,4',5',6-Hepta-CB	191	5.0	2.5	0.5	0.25	50.0
2,3,3',4,5,5',6-Hepta-CB	192	5.0	2.5	0.5	0.25	50.0
2,3,3',4',5,5',6-Hepta-CB	193	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,4',5,5'-OctaCB	194	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,4',5,6-OctaCB	195	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,4',5,6'-OctaCB 2,2',3,4,4',5,5',6-OctaCB	196/203	10.0	5.0	1.0	0.50	100.0
2,2',3,3',4,4',6,6'-Octa-CB	197	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,5,5',6-Octa-CB	198	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,5,5',6'-Octa-CB	199	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,5,6,6'-Octa-CB	200	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,5',6,6'-Octa-CB	201	5.0	2.5	0.5	0.25	50.0
2,2',3,3'5,5',6,6'-Octa-CB	202	5.0	2.5	0.5	0.25	50.0
2,2',3,4,4',5,6,6'-Octa-CB	204	5.0	2.5	0.5	0.25	50.0
2,3,3',4,4',5,5',6-Octa-CB	205	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,4',5,5',6-No-CB	206	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,4',5,6,6'-No-CB	207	5.0	2.5	0.5	0.25	50.0
2,2',3,3',4,5,5',6,6'-No-CB	208	5.0	2.5	0.5	0.25	50.0
Dec-CB	209	5.0	2.5	0.5	0.25	50.0

Total PCB Homologues						
Total PCB Homologues		**Water (pg/L)**	**Solid (pg/g)**	**Fish/Tissue (pg/g)**		
Monochlorobiphenyl		5.0	2.5	0.5		
Dichlorobiphenyl		5.0	2.5	0.5		
Trichlorobiphenyl		5.0	2.5	0.5		
Tetrachlorobiphenyl		5.0	2.5	0.5		
Pentachlorobiphenyl		5.0	2.5	0.5		

Table 1

PCB Target Compounds and Quantitation Limits

Hexachlorobiphenyl		5.0	2.5	0.5		
Heptachlorobiphenyl		5.0	2.5	0.5		
Octachlorobiphenyl		5.0	2.5	0.5		
Nonachlorobiphenyl		5.0	2.5	0.5		
Decachlorobiphenyl		5.0	2.5	0.5		

- Quantitation limits listed are based upon 1 liter of aqueous sample, 10 grams dry weight solid, 10 grams fish/tissue sample and 20 grams Sample size for Serum [Serum (pg/g-Lipids) assumes 0.5% Lipid content]. Quantitation limits for co-eluting congeners are based on their sum.
- Final Volumes for all matrices are 20 μL except Solid is 100 μL.
- *California SIP samples have a reporting limit of 20 pg/L for all congeners.*
- For DOD clients the lowest standard of the calibration establishes the QL.

Table 2

Concentration of PCBs in Calibration and Calibration Verification Solutions (pg/μL)

2a. PCB Congener	Congener No.	CS0	CS1	CS2	CS3*	CS4	CS5
2-Mono-CB	1	0.25	1.0	2.5	50	400	1000
3-Mono-CB	2	0.25	1.0	2.5	50	400	1000
4-Mono-CB	3	0.25	1.0	2.5	50	400	1000
2,2'-Di-CB 2,6-Di-CB	4/10	0.5	2.0	5.0	100	800	2000
2,3'-Di-CB	6	0.25	1.0	2.5	50	400	1000
2,3-Di-CB 2,4'-Di-CB	5/8	0.5	2.0	5.0	100	800	2000
2,4-Di-CB 2,5-Di-CB	7/9	0.5	2.0	5.0	100	800	2000
3,3'-Di-CB	11	0.25	1.0	2.5	50	400	1000
3,4-Di-CB 3,4'-Di-CB	12/13	0.5	2.0	5.0	100	800	2000
3,5-Di-CB	14	0.25	1.0	2.5	50	400	1000
4,4'-Di-CB	15	0.25	1.0	2.5	50	400	1000
2,2',3-Tri-CB 2,4',6-Tri-CB	16/32	0.5	2.0	5.0	100	800	2000
2,2',4-Tri-CB	17	0.25	1.0	2.5	50	400	1000
2,2',5-Tri-CB	18	0.25	1.0	2.5	50	400	1000
2,2',6-Tri-CB	19	0.25	1.0	2.5	50	400	1000
2,3,3'-Tri-CB 2,3,4-Tri-CB 2',3,4-Tri-CB	20/21/33	0.75	3.0	7.5	150	1200	3000
2,3,4'-Tri-CB	22	0.25	1.0	2.5	50	400	1000
2,3,5-Tri-CB	23	0.25	1.0	2.5	50	400	1000
2,3,6-Tri-CB 2,3',6-Tri-CB	24/27	0.5	2.0	5.0	100	800	2000
2,3',4-Tri-CB	25	0.25	1.0	2.5	50	400	1000
2,3',5-Tri-CB	26	0.25	1.0	2.5	50	400	1000
2,4,4'-Tri-CB	28	0.25	1.0	2.5	50	400	1000
2,4,5-Tri-CB	29	0.25	1.0	2.5	50	400	1000
2,4,6-Tri-CB	30	0.25	1.0	2.5	50	400	1000
2,4',5-Tri-CB	31	0.25	1.0	2.5	50	400	1000
2',3,5-Tri-CB	34	0.25	1.0	2.5	50	400	1000
3,3',4-Tri-CB	35	0.25	1.0	2.5	50	400	1000
3,3',5-Tri-CB	36	0.25	1.0	2.5	50	400	1000
3,4,4'-Tri-CB	37	0.25	1.0	2.5	50	400	1000
3,4,5-Tri-CB	38	0.25	1.0	2.5	50	400	1000
3,4',5-Tri-CB	39	0.25	1.0	2.5	50	400	1000
2,2',3,3'-Tetra-CB	40	0.25	1.0	2.5	50	400	1000

Table 2

Concentration of PCBs in Calibration and Calibration Verification Solutions (pg/μL)

2a. PCB Congener	Congener No.	CS0	CS1	CS2	CS3*	CS4	CS5
2,2',3,4-Tetra-CB 2,3,4',6-Tetra-CB 2,3',4',6-Tetra-CB 2,3',5,5'-Tetra-CB	41/64/71/72	1.0	4.0	10	200	1600	4000
2,3',5,5'-Tetra-CB 2,2',3,4'-Tetra-CB	42/59	0.50	2.0	5.0	100	800	2000
2,3,3',6-Tetra-CB 2,2',3,5-Tetra-CB	43/49	0.50	2.0	5.0	100	800	2000
2,2',4,5'-Tetra-CB	44	0.25	1.0	2.5	50	400	1000
2,2',3,5'-Tetra-CB	45	0.25	1.0	2.5	50	400	1000
2,2',3,6-Tetra-CB	46	0.25	1.0	2.5	50	400	1000
2,2',3,6'-Tetra-CB	47	0.25	1.0	2.5	50	400	1000
2,2',4,4'-Tetra-CB 2,2',4,5-Tetra-CB	48/75	0.50	2.0	5.0	100	800	2000
2,4,4',6-Tetra-CB	50	0.25	1.0	2.5	50	400	1000
2,2',4,6-Tetra-CB	51	0.25	1.0	2.5	50	400	1000
2,2',4,6'-Tetra-CB 2,2',5,5'-Tetra-CB	52/69	0.50	2.0	5.0	100	800	2000
2,3',4,6-Tetra-CB	53	0.25	1.0	2.5	50	400	1000
2,2',5,6'-Tetra-CB	54	0.25	1.0	2.5	50	400	1000
2,2',6,6'-Tetra-CB	55	0.25	1.0	2.5	50	400	1000
2,3,3',4-Tetra-CB 2,3,3',4'-Tetra-CB	56/60	0.50	2.0	5.0	100	800	2000
2,3,4,4'-Tetra-CB	57	0.25	1.0	2.5	50	400	1000
2,3,3',5-Tetra-CB	58	0.25	1.0	2.5	50	400	1000
2,3,4,5-Tetra-CB 2,3',4',5-Tetra-CB	61/70	0.50	2.0	5.0	100	800	2000
2,3',4',5-Tetra-CB	62	0.25	1.0	2.5	50	400	1000
2,3,4,6-Tetra-CB	63	0.25	1.0	2.5	50	400	1000
2,3,4',5-Tetra-CB	65	0.25	1.0	2.5	50	400	1000
2,3,5,6-Tetra-CB	67	0.25	1.0	2.5	50	400	1000
2,3',4,5-Tetra-CB	68	0.25	1.0	2.5	50	400	1000
2,3',4',5-Tetra-CB	73	0.25	1.0	2.5	50	400	1000
2,3',5',6-Tetra-CB	74	0.25	1.0	2.5	50	400	1000
2,4,4',5-Tetra-CB 2',3,4,5-Tetra-CB	76/66	0.50	2.0	5.0	100	800	2000
2,3',4,4'-Tetra-CB	77	0.25	1.0	2.5	50	400	1000
3,3',4,4'-Tetra-CB	78	0.25	1.0	2.5	50	400	1000
3,3',4,5-Tetra-CB	79	0.25	1.0	2.5	50	400	1000
3,3',4,5'-Tetra-CB	80	0.25	1.0	2.5	50	400	1000
3,3',5,5'-Tetra-CB	81	0.25	1.0	2.5	50	400	1000

Table 2

Concentration of PCBs in Calibration and Calibration Verification Solutions (pg/μL)

2a. PCB Congener	Congener No.	CS0	CS1	CS2	CS3*	CS4	CS5
3,4,4',5-Tetra-CB	82	0.25	1.0	2.5	50	400	1000
2,2',3,3',4-Penta-CB	83	0.25	1.0	2.5	50	400	1000
2,2',3,3',5-Penta-CB 2,2',3,3',6-Penta-CB	84/92	0.50	2.0	5.0	100	800	2000
2,2',3,5,5'-Penta-CB 2,2',3,4,4'-Penta-CB	85/116	0.50	2.0	5.0	100	800	2000
2,3,4,5,6-Penta-CB	86	0.25	1.0	2.5	50	400	1000
2,2',3,4,5-Penta-CB 2,2',3,4,5'-Penta-CB 2,3,4',5,6-Penta-CB	87/117/125	0.75	3.0	7.5	150	1200	3000
2',3,4,5,6'-Penta-CB 2,2',3,4,6-Penta-CB	88/91	0.50	2.0	5.0	100	800	2000
2,2',3,4',6-Penta-CB	89	0.25	1.0	2.5	50	400	1000
2,2',3,4,6'-Penta-CB 2,2',3,4',5-Penta-CB	90/101	0.50	2.0	5.0	100	800	2000
2,2',4,5,5'-Penta-CB	93	0.25	1.0	2.5	50	400	1000
2,2',3,5,6-Penta-CB	94	0.25	1.0	2.5	50	400	1000
2,2',3,5,6'-Penta-CB 2,2',3,5',6-Penta-CB 2,2',3',4,6-Penta-CB	95/98/102	0.75	3.0	7.5	150	1200	3000
2,2',4,5,6'-Penta-CB	96	0.25	1.0	2.5	50	400	1000
2,2',3,6,6'-Penta-CB	97	0.25	1.0	2.5	50	400	1000
2,2',3,4',5-Penta-CB	99	0.25	1.0	2.5	50	400	1000
2,2',4,4',5-Penta-CB	100	0.25	1.0	2.5	50	400	1000
2,2',4,4',6-Penta-CB	103	0.25	1.0	2.5	50	400	1000
2,2',4,5',6-Penta-CB	104	0.25	1.0	2.5	50	400	1000
2,2',4,4,6'-Penta-CB	105	0.25	1.0	2.5	50	400	1000
2,3',4,4',5-Penta-CB 2,3,3',4,5-Penta-CB	118/106	0.50	2.0	5.0	100	800	2000
2,3',4,4',5-Penta-CB 2,3,3',4',5-Penta-CB	107/109	0.50	2.0	5.0	100	800	2000
2,3,3',4,6-Penta-CB 2,3,3',4,5'-Penta-CB	108/112	0.50	2.0	5.0	100	800	2000
2,3,3',5,6-Penta-CB	110	0.25	1.0	2.5	50	400	1000
2,3,3',4',6-Penta-CB 2,3,3',5,5'-Penta-CB	111/115	0.50	2.0	5.0	100	800	2000
2,3,4,4',6-Penta-CB	113	0.25	1.0	2.5	50	400	1000
2,3,4,4',5-Penta-CB	114	0.25	1.0	2.5	50	400	1000
2,3,4,4',5-Penta-CB	119	0.25	1.0	2.5	50	400	1000
2,3',4,4',6-Penta-CB	120	0.25	1.0	2.5	50	400	1000
2,3',4,5,5'-Penta-CB	121	0.25	1.0	2.5	50	400	1000

Table 2

Concentration of PCBs in Calibration and Calibration Verification Solutions (pg/μL)

2a. PCB Congener	Congener No.	CS0	CS1	CS2	CS3*	CS4	CS5
2',3,3'4,5-Penta-CB	122	0.25	1.0	2.5	50	400	1000
2',3,4,4',5-Penta-CB	123	0.25	1.0	2.5	50	400	1000
2',3,4,5,5'-Penta-CB	124	0.25	1.0	2.5	50	400	1000
3,3',4,4',5-Penta-CB	126	0.25	1.0	2.5	50	400	1000
3,3'4,4',5-Penta-CB	127	0.25	1.0	2.5	50	400	1000
3,3',4,5,5'-Penta-CB 2,2',3,3',4,4'-Hexa-CB	128/162	0.50	2.0	5.0	100	800	2000
2,3,3',4',5,5'-Hexa-CB	129	0.25	1.0	2.5	50	400	1000
2,2',3,3',4,5-Hexa-CB	130	0.25	1.0	2.5	50	400	1000
2,2'3,3'4,6-Hexa-CB 2,2',3,3',5,5'-Hexa-CB	131/133	0.50	2.0	5.0	100	800	2000
2,3,3',4,5',6-Hexa-CB 2,2',3,3',5,5'-Hexa-CB	132/161	0.50	2.0	5.0	100	800	2000
2,2',3,4,5,6-Hexa-CB 2,2',3,3',5,6-Hexa-CB	134/143	0.50	2.0	5.0	100	800	2000
2,2',3,3',5,6'-Hexa-CB	135	0.25	1.0	2.5	50	400	1000
2,2',3,3',5,6'-Hexa-CB	136	0.25	1.0	2.5	50	400	1000
2,2',3,3',6,6'-Hexa-CB	137	0.25	1.0	2.5	50	400	1000
2,2',3,4,4',5-Hexa-CB 2,2',3,4,4',5'-Hexa-CB 2,3,3',4',5,6-Hexa-CB	138/163/164	0.75	3.0	7.5	150	1200	3000
2,3,3',4',5',6-Hexa-CB 2,2',3,4,4',6-Hexa-CB	139/149	0.50	2.0	5.0	100	800	2000
2,2',3,4',5',6-Hexa-CB	140	0.25	1.0	2.5	50	400	1000
2,2',3,4,4',6'-Hexa-CB	141	0.25	1.0	2.5	50	400	1000
2,2',3,4,5,6-Hexa-CB	142	0.25	1.0	2.5	50	400	1000
2,2',3,4,5,5'-Hexa-CB	144	0.25	1.0	2.5	50	400	1000
2,2',3,4,5',6-Hexa-CB	145	0.25	1.0	2.5	50	400	1000
2,2',3,4,6,6'-Hexa-CB 2,2',3,4',5,5'-Hexa-CB	146/165	0.50	2.0	5.0	100	800	2000
2,3,3',5,5',6-Hexa-CB	147	0.25	1.0	2.5	50	400	1000
2,2',3,4',5,6-Hexa-CB	148	0.25	1.0	2.5	50	400	1000
2,2',3,4',5,6'-Hexa-CB	150	0.25	1.0	2.5	50	400	1000
2,2',3,4',6,6'-Hexa-CB	151	0.25	1.0	2.5	50	400	1000
2,2',3,5,5',6-Hexa-CB	152	0.25	1.0	2.5	50	400	1000
2,2',3,5,6,6'-Hexa-CB	153	0.25	1.0	2.5	50	400	1000
2,2'4,4',5',6-Hexa-CB	154	0.25	1.0	2.5	50	400	1000
2,2',4,4',5,6'-Hexa-CB	155	0.25	1.0	2.5	50	400	1000
2,2',4,4',6,6'-Hexa-CB	156	0.25	1.0	2.5	50	400	1000
2,3,3',4,4',5-Hexa-CB	157	0.25	1.0	2.5	50	400	1000

Table 2

Concentration of PCBs in Calibration and Calibration Verification Solutions (pg/μL)

2a. PCB Congener	Congener No.	CS0	CS1	CS2	CS3*	CS4	CS5
2,3,3',4,4',5'-Hexa-CB 2,3,3',5,5',6-Hexa-CB	158/160	0.50	2.0	5.0	100	800	2000
2,3,3',4,5,6-Hexa-CB	159	0.25	1.0	2.5	50	400	1000
2,3,3',4,5,5'-Hexa-CB	166	0.25	1.0	2.5	50	400	1000
2,3',4,4',5,5'-Hexa-CB	167	0.25	1.0	2.5	50	400	1000
2,3',4,4',5,5'-Hexa-CB	168	0.25	1.0	2.5	50	400	1000
3,3',4,4',5,5'-Hexa-CB	169	0.25	1.0	2.5	50	400	1000
3,3',4,4',5,5'-Hexa-CB	170	0.25	1.0	2.5	50	400	1000
2,2',3,3',4,4',5-Hepta-CB	171	0.25	1.0	2.5	50	400	1000
2,2',3,3',4,4',6-Hepta-CB	174	0.25	1.0	2.5	50	400	1000
2,2',3,3',4,5,5'-Hepta-CB	172	0.25	1.0	2.5	50	400	1000
2,2',3,3',4,5,6-Hepta-CB	173	0.25	1.0	2.5	50	400	1000
2,2',3,3',4,5,6'-Hepta-CB	174	0.25	1.0	2.5	50	400	1000
2,2',3,3',4,5',6-Hepta-CB	175	0.25	1.0	2.5	50	400	1000
2,2',3,3',4,6,6'-Hepta-CB	176	0.25	1.0	2.5	50	400	1000
2,2',3,3',4',5,6-Hepta-CB	177	0.25	1.0	2.5	50	400	1000
2,2',3,3',5,5',6-Hepta-CB	178	0.25	1.0	2.5	50	400	1000
2,2',3,3',5,6,6'-Hepta-CB	179	0.25	1.0	2.5	50	400	1000
2,2',3,4,4',5,5'-Hepta-CB	180	0.25	1.0	2.5	50	400	1000
2,2',3,4,4',5,6-Hepta-CB	181	0.25	1.0	2.5	50	400	1000
2,2',3,4,4',5,6'-Hepta-CB 2,2',3,4',5,5',6-Hepta-CB	182/187	0.50	2.0	5.0	100	800	2000
2,2',3,4,4',5',6-Hepta-CB	183	0.25	1.0	2.5	50	400	1000
2,2',3,4,4',6,6'-Hepta-CB	184	0.25	1.0	2.5	50	400	1000
2,2',3,4,5,5',6-Hepta-CB	185	0.25	1.0	2.5	50	400	1000
2,2',3,4,5,6,6'Hepta-CB	186	0.25	1.0	2.5	50	400	1000
2,2',3,4',5,6,6'-Hepta-CB	188	0.25	1.0	2.5	50	400	1000
2,3,3',4,4',5,5'-Hepta-CB	189	0.25	1.0	2.5	50	400	1000
2,3,3',4,4',5,6-Hepta-CB	190	0.25	1.0	2.5	50	400	1000
2,3,3',4,4',5',6-Hepta-CB	191	0.25	1.0	2.5	50	400	1000
2,3,3',4,5,5',6-Hepta-CB	192	0.25	1.0	2.5	50	400	1000
2,3,3',4',5,5',6-Hepta-CB	193	0.25	1.0	2.5	50	400	1000
2,2',3,3',4,4',5,5'-Octa-CB	194	0.25	1.0	2.5	50	400	1000
2,2',3,3',4,4',5,6-Octa-CB	195	0.25	1.0	2.5	50	400	1000
2,2',3,3',4,4',5,6'-Octa-CB 2,2',3,4,4',5,5',6-Octa-CB	196/203	0.50	2.0	5.0	100	800	2000
2,2',3,3',4,4',6,6'-Octa-CB	197	0.25	1.0	2.5	50	400	1000
2,2',3,3',4,5,5',6-Octa-CB	198	0.25	1.0	2.5	50	400	1000
2,2',3,3',4,5,5',6'-Octa-CB	199	0.25	1.0	2.5	50	400	1000
2,2',3,3',4,5,6,6'-Octa-CB	200	0.25	1.0	2.5	50	400	1000
2,2',3,3',4,5',6,6'-Octa-CB	201	0.25	1.0	2.5	50	400	1000

Table 2

Concentration of PCBs in Calibration and Calibration Verification Solutions (pg/μL)

2a. PCB Congener	Congener No.	CS0	CS1	CS2	CS3*	CS4	CS5
2,2',3,3'5,5',6,6'-Octa-CB	202	0.25	1.0	2.5	50	400	1000
2,2',3,4,4',5,6,6'-Octa-CB	204	0.25	1.0	2.5	50	400	1000
2,3,3',4,4',5,5',6-Octa-CB	205	0.25	1.0	2.5	50	400	1000
2,2',3,3',4,4',5,5',6-No-CB	206	0.25	1.0	2.5	50	400	1000
2,2',3,3',4,4',5,6,6'-No-CB	207	0.25	1.0	2.5	50	400	1000
2,2',3,3',4,5,5',6,6'-No-CB	208	0.25	1.0	2.5	50	400	1000
De-CB	209	0.25	1.0	2.5	50	400	1000

*Calibration Verification Solution

Table 2

Concentration of labeled PCBs in Calibration and Calibration Verification Solutions (pg/μL)

2b. Internal Standards	Congener	CS1	CS2	CS3*	CS4	CS5
^{13}C-2-Mono-CB	1	100	100	100	100	100
^{13}C-4-Mono-CB	3	100	100	100	100	100
^{13}C-2,2'-Di-CB	4	100	100	100	100	100
^{13}C-2,5-Di-CB	9	100	100	100	100	100
^{13}C-3,3'-Di-CB	11	100	100	100	100	100
^{13}C-2,2',6-Tri-CB	19	100	100	100	100	100
^{13}C-2,4,4'-Tri-CB	28	100	100	100	100	100
^{13}C-2,4',6-Tri-CB	32	100	100	100	100	100
^{13}C-3,4,4'-Tri-CB	37	100	100	100	100	100
^{13}C-2,2',4,4'-Tetra-CB	47	100	100	100	100	100
^{13}C-2,2',4,6'-Tetra-CB	52	100	100	100	100	100
^{13}C-2,2',6,6'-Tetra-CB	54	100	100	100	100	100
^{13}C-2,3',4',5-Tetra-CB	70	100	100	100	100	100
^{13}C-3,3',4,4'-Tetra-CB	77	100	100	100	100	100
^{13}C -3,3',5,5'-Tetra-CB	80	100	100	100	100	100
^{13}C-3,4,4',5'-Tetra-CB	81	100	100	100	100	100
^{13}C-2,2',3,5',6-Penta-CB	95	100	100	100	100	100
^{13}C-2,2',3,4',5'-Penta-CB	97	100	100	100	100	100
^{13}C-2,2',4,5,5'-Penta-CB	101	100	100	100	100	100
^{13}C-2,2',4,4,6'-Penta-CB	104	100	100	100	100	100
^{13}C-2,3,3',4,4'-Penta-CB	105	100	100	100	100	100
^{13}C-2,3,4,4',5-Penta-CB	114	100	100	100	100	100
^{13}C-2,3',4,4',5-Penta-CB	118	100	100	100	100	100

Table 2

Concentration of labeled PCBs in Calibration and Calibration Verification Solutions (pg/μL)

2b. Internal Standards	Congener	CS1	CS2	CS3*	CS4	CS5
^{13}C-2',3,4,4',5-Penta-CB	123	100	100	100	100	100
^{13}C-3,3',4,4',5-Penta-CB	126	100	100	100	100	100
^{13}C-3,3',4,5,5'-Penta-CB	127	100	100	100	100	100
^{13}C-2,2',3,4,4',5'-Hexa-CB	138	100	100	100	100	100
^{13}C-2,2',3,4,5,5'-Hexa-CB	141	100	100	100	100	100
^{13}C-2,2',4,4',5,5'-Hexa-CB	153	100	100	100	100	100
^{13}C-2,2',4,4',6,6'-Hexa-CB	155	100	100	100	100	100
^{13}C-2,3,3',4,4',5-Hexa-CB	156	100	100	100	100	100
^{13}C-2,3,3',4,4',5'-Hexa-CB	157	100	100	100	100	100
^{13}C-2,3,3',4,5,5'-Hexa-CB	159	100	100	100	100	100
^{13}C-2,3',4,4',5,5'-Hexa-CB	167	100	100	100	100	100
^{13}C-3,3',4,4',5,5'-Hexa-CB	169	100	100	100	100	100
^{13}C-2,2',3,3',4,4',5-Hepta-CB	170	100	100	100	100	100
^{13}C-2,2',3,4,4',5,5'-Hepta-CB	180	100	100	100	100	100
^{13}C-2,2',3,4',5,6,6'-Hepta-CB	188	100	100	100	100	100
^{13}C-2,3,3',4,4',5,5'-Hepta-CB	189	100	100	100	100	100
^{13}C-2,2',3,3',4,4',5,5'-Octa-CB	194	100	100	100	100	100
^{13}C-2,2',3,3',5,5',6,6'-Octa-CB	202	100	100	100	100	100
^{13}C-2,2',3,3',4,4',5,5',6-Nona-CB	206	100	100	100	100	100
^{13}C-2,2',3,3',4,5,5',6,6'-Nona-CB	208	100	100	100	100	100
^{13}C-Deca-CB	209	100	100	100	100	100
2c. Recovery Standards						
^{13}C-4,4'-Di-CB	15	100	100	100	100	100
^{13}C-2,4',5-Tri-CB	31	100	100	100	100	100
^{13}C-2,3,4,4'-Tetra-CB	60	100	100	100	100	100
^{13}C-2,3,3',5,5'-Penta-CB	111	100	100	100	100	100
^{13}C-2,2',3,4,4,',5'-Hexa-CB	128	100	100	100	100	100
^{13}C-2,3,3',4,4',5,5',6-Octa-CB	205	100	100	100	100	100
2d. Cleanup Recovery Standards						
^{13}C-2,2',5,5'-Tetra-CB	79	100	100	100	100	100
^{13}C-2,2,3,3,5,5',6-Hepta-CB	178	100	100	100	100	100

Table 3

Theoretical Ion Abundance Ratios and QC Limits

Number of Chlorine Atoms	Ion Type	Theoretical Ratio	Control Limits[1]	
			Lower	Upper
1	M/M+2	3.13	2.66	3.60
2	M/M+2	1.56	1.33	1.79
3	M/M+2	1.04	0.88	1.20
4	M/M+2	0.77	0.65	0.89
5	M+2/M+4	1.55	1.32	1.78
6	M+2/M+4	1.24	1.05	1.43
7	M+2/M+4	1.05	0.89	1.21
7[1]	M/M+2	0.45	0.38	0.52
8	M+2/M+4	0.89	0.76	1.02
9	M+4/M+6	1.34	1.14	1.54
9[2]	M+2/M+4	0.77	0.65	0.89
10	M+4/M+6	1.16	0.99	1.33

(1) Used for 13C-Hepa-CB
(2) Used for 13C-Nona-CB

Table 4

Quantitation Limits for the Determination of Coplanar/Mono-Ortho PCBs Only

Coplanar/Mono-Ortho PCB Target Compounds				
Target Compound	Congener Number	QL Water (pg/L)	QL Solid (pg/g)	QL Fish/Tissue (pg/g)
3,3',4,4'-Tetra-CB	77	5.0	1.25	0.5
3,4,4',5-Tetra-CB	81	5.0	1.25	0.5
2,3,3',4,4'-Penta-CB	105	5.0	1.25	0.5
2,3,4,4',5-Penta-CB	114	5.0	1.25	0.5
2,3',4,4',5-Penta-CB 2,3,3',4,5-Penta-CB	118/106	10	2.5	1.0
2',3,4,4',5-Penta-CB	123	5.0	1.25	0.5
3,3',4,4'5-Penta-CB	126	5.0	1.25	0.5
2,3,3',4,4',5-Hexa-CB	156	5.0	1.25	0.5
2,3,3',4,4',5'-Hexa-CB	157	5.0	1.25	0.5
2,3',4,4',5,5'-Hexa-CB	167	5.0	1.25	0.5
3,3',4,4',5,5'-Hexa-CB	169	5.0	1.25	0.5
2,3,3',4,4',5,5'-Hepta-CB	189	5.0	1.25	0.5

- Quantitation limits are based on 1 liter aqueous sample, 10 grams dry weight solid, and 10 grams fish/tissue sample. Final volume of 20µl for water and tissue and 50µl for solid matrices.

Table 5

Concentration of Coplanar/Mono-Ortho PCBs in Calibration Solutions

		Solution Concentration (pg/μL)					
Coplanar/Mono-Ortho PCB Congeners	**Congener No.**	**CS0**	**CS1**	**CS2**	**CS3***	**CS4**	**CS5**
3,3',4,4'-Tetra-CB	77	0.25	1.0	2.5	50	400	1000
3,4,4',5-Tetra-CB	81	0.25	1.0	2.5	50	400	1000
2,3,3',4,4'-Penta-CB	105	0.25	1.0	2.5	50	400	1000
2,3,4,4',5-Penta-CB	114	0.25	1.0	2.5	50	400	1000
2,3',4,4',5-Penta-CB 2,3,3',4,5-Penta-CB	118/106	1.0	2.0	5.0	100	800	2000
2',3,4,4',5-Penta-CB	123	0.25	1.0	2.5	50	400	1000
3,3',4,4',5-Penta-CB	126	0.25	1.0	2.5	50	400	1000
2,3,3',4,4',5-Hexa-CB	156	0.25	1.0	2.5	50	400	1000
2,3,3',4,4',5'-Hexa-CB	157	0.25	1.0	2.5	50	400	1000
2,3',4,4',5,5'-Hexa-CB	167	0.25	1.0	2.5	50	400	1000
3,3',4,4',5,5'-Hexa-CB	169	0.25	1.0	2.5	50	400	1000
2,3,3',4,4',5,5'-Hepta-CB	189	0.25	1.0	2.5	50	400	1000
Internal Standards							
^{13}C-3,3',4,4'-Tetra-CB	77	100	100	100	100	100	100
^{13}C-3,4,4',5'-Tetra-CB	81	100	100	100	100	100	100
^{13}C-2,3,3',4,4'-Penta-CB	105	100	100	100	100	100	100
^{13}C-2,3,4,4',5-Penta-CB	114	100	100	100	100	100	100
^{13}C-2,3',4,4',5-Penta-CB	118	100	100	100	100	100	100
^{13}C-2',3,4,4',5-Penta-CB	123	100	100	100	100	100	100
^{13}C-3,3',4,4',5-Penta-CB	126	100	100	100	100	100	100
^{13}C-2,3,3',4,4',5-Hexa-CB	156	100	100	100	100	100	100
^{13}C-2,3,3',4,4',5'-Hexa-CB	157	100	100	100	100	100	100
^{13}C-2,3,3',4,4',5'-Hexa-CB	167	100	100	100	100	100	100
^{13}C-3,3',4,4',5,5'-Hexa-CB	169	100	100	100	100	100	100
^{13}C-2,3,3',4,4',5,5'-Hepta-CB	189	100	100	100	100	100	100
Recovery Standards							
^{13}C-2,3,3',4'-Tetra-CB	60	100	100	100	100	100	100
^{13}C-2,3,3',5,5'-Penta-CB (1)	111	100	100	100	100	100	100
^{13}C-2,2',3,3',4,4'-Hexa-CB	128	100	100	100	100	100	100

* Calibration Verification Solution

(1) Used only if interferences are present

Table 6

Exact Masses Monitored for PCBs

Compound	Native PCBs	Internal Standard PCBs
Mono-CB	188.0393, 190.0363	200.0795, 202.0766
Di-CB	222.0003, 223.9974	234.0406, 236.0376
Tri-CB	255.9613, 257.9584	268.0016, 269.9986
Tetra-CB	289.9224, 291.9194	301.9626, 303.9597
Penta-CB	325.8804, 327.8775	337.9207, 339.9177
Hexa-CB	359.8415, 361.8385	371.8817, 373.8788
Hepta-CB	393.8025, 395.7995	403.8457, 405.8428
Octa-CB	427.7635, 429.7606	439.8038, 441.8008
Nona-CB	463.7216, 465.7186	473.7648, 475.7619
Deca-CB	497.6826, 499.6797	509.7229, 511.7199

Table 7
Acceptance Criteria for Concentrations of PCBs in QC

	1668A				1668C		
Congener	**Congener No.**	**VER (%)**	**OPR (%)**	**IS Samples(%)**	**VER (%)**	**OPR (%)**	**IS Samples(%)**
2-Mono-CB	1	70-130	50-150	NA	75-125	60-135	NA
3-Mono-CB	2	70-130	50-150	NA	75-125	60-135	NA
4-Mono-CB	3	70-130	50-150	NA	75-125	60-135	NA
2,2'-Di-CB 2,6-Di-CB	4/10	70-130	50-150	NA	75-125	60-135	NA
2,3-Di-CB 2,4'-Di-CB6	5/8	70-130	50-150	NA	75-125	60-135	NA
2,3'-Di-CB	6	70-130	50-150	NA	75-125	60-135	NA
2,4-Di-CB 2,5-Di-CB	7/9	70-130	50-150	NA	75-125	60-135	NA
3,3'-Di-CB	11	70-130	50-150	NA	75-125	60-135	NA
3,4-Di-CB 3,4'-Di-CB	12/13	70-130	50-150	NA	75-125	60-135	NA
3,5-Di-CB	14	70-130	50-150	NA	75-125	60-135	NA
4,4'-Di-CB	15	70-130	50-150	NA	75-125	60-135	NA
2,2',3-Tri-CB 2,4',6-Tri-CB	16/32	70-130	50-150	NA	75-125	60-135	NA
2,2',4-Tri-CB	17	70-130	50-150	NA	75-125	60-135	NA
2,2',5-Tri-CB	18	70-130	50-150	NA	75-125	60-135	NA
2,2',6-Tri-CB	19	70-130	50-150	NA	75-125	60-135	NA
2,3,3'-Tri-CB 2,3,4-Tri-CB 2',3,4-Tri-CB	20/21/33	70-130	50-150	NA	75-125	60-135	NA
2,3,4'-Tri-CB	22	70-130	50-150	NA	75-125	60-135	NA
2,3,5-Tri-CB	23	70-130	50-150	NA	75-125	60-135	NA
2,3,6-Tri-CB 2,3',6-Tri-CB	24/27	70-130	50-150	NA	75-125	60-135	NA
2,3',4-Tri-CB	25	70-130	50-150	NA	75-125	60-135	NA
2,3',5-Tri-CB	26	70-130	50-150	NA	75-125	60-135	NA
2,4,4'-Tri-CB	28	70-130	50-150	NA	75-125	60-135	NA
2,4,5-Tri-CB	29	70-130	50-150	NA	75-125	60-135	NA
2,4,6-Tri-CB	30	70-130	50-150	NA	75-125	60-135	NA
2,4',5-Tri-CB	31	70-130	50-150	NA	75-125	60-135	NA
2',3,5-Tri-CB	34	70-130	50-150	NA	75-125	60-135	NA
3,3',4-Tri-CB	35	70-130	50-150	NA	75-125	60-135	NA
3,3',5-Tri-CB	36	70-130	50-150	NA	75-125	60-135	NA
3,4,4'-Tri-CB	37	70-130	50-150	NA	75-125	60-135	NA
3,4,5-Tri-CB	38	70-130	50-150	NA	75-125	60-135	NA
3,4',5-Tri-CB	39	70-130	50-150	NA	75-125	60-135	NA

Table 7
Acceptance Criteria for Concentrations of PCBs in QC

	1668A				1668C		
Congener	**Congener No.**	**VER (%)**	**OPR (%)**	**IS Samples(%)**	**VER (%)**	**OPR (%)**	**IS Samples(%)**
2,2’,3,3’-Tetra-CB	40	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4-Tetra-CB 2,3,4',6-Tetra-CB 2,3',4',6-Tetra-CB 2,3',5,5'-Tetra-CB	41/64/71/72	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4'-Tetra-CB 2,3,3',6-Tetra-CB	42/59	70-130	50-150	NA	75-125	60-135	NA
2,2',3,5-Tetra-CB 2,2',4,5'-Tetra-CB	43/49	70-130	50-150	NA	75-125	60-135	NA
2,2’,3,5’-Tetra-CB	44	70-130	50-150	NA	75-125	60-135	NA
2,2’,3,6-Tetra-CB‘	45	70-130	50-150	NA	75-125	60-135	NA
2,2’,3,6’-Tetra-CB	46	70-130	50-150	NA	75-125	60-135	NA
2,2’,4,4’-Tetra-CB	47	70-130	50-150	NA	75-125	60-135	NA
2,2',4,5-Tetra-CB 2,4,4',6-Tetra-CB	48/75	70-130	50-150	NA	75-125	60-135	NA
2,2’,4,6-Tetra-CB	50	70-130	50-150	NA	75-125	60-135	NA
2,2’,4,6’-Tetra-CB	51	70-130	50-150	NA	75-125	60-135	NA
2,2',5,5'-Tetra-CB 2,3',4,6-Tetra-CB	52/69	70-130	50-150	NA	75-125	60-135	NA
2,2’,5,6’-Tetra-CB	53	70-130	50-150	NA	75-125	60-135	NA
2,2’,6,6’-Tetra-CB	54	70-130	50-150	NA	75-125	60-135	NA
2,3,3’,4-Tetra-CB	55	70-130	50-150	NA	75-125	60-135	NA
2,3,3',4'-Tetra-CB 2,3,4,4'-Tetra-CB	56/60	70-130	50-150	NA	75-125	60-135	NA
2,3,3’,5-Tetra-CB	57	70-130	50-150	NA	75-125	60-135	NA
2,3,3’,5’-Tetra-CB	58	70-130	50-150	NA	75-125	60-135	NA
2,3,4,5-Tetra-CB 2,3',4',5-Tetra-CB	61/70	70-130	50-150	NA	75-125	60-135	NA
2,3,4,6-Tetra-CB	62	70-130	50-150	NA	75-125	60-135	NA
2,3,4’,5-Tetra-CB	63	70-130	50-150	NA	75-125	60-135	NA
2,3,5,6-Tetra-CB	65	70-130	50-150	NA	75-125	60-135	NA
2,3’,4,5-Tetra-CB	67	70-130	50-150	NA	75-125	60-135	NA
2,3’,4,5’-Tetra-CB	68	70-130	50-150	NA	75-125	60-135	NA
2,3’,5’,6-Tetra-CB	73	70-130	50-150	NA	75-125	60-135	NA
2,4,4’,5-Tetra-CB	74	70-130	50-150	NA	75-125	60-135	NA
2',3,4,5-Tetra-CB 2,3',4,4'-Tetra-CB	76/66	70-130	50-150	NA	75-125	60-135	NA
3,3’,4,5-Tetra-CB	77	70-130	50-150	NA	75-125	60-135	NA
3,3’,4,5’-Tetra-CB	78	70-130	50-150	NA	75-125	60-135	NA
3,3’,5,5’-Tetra-CB	79	70-130	50-150	NA	75-125	60-135	NA

Table 7
Acceptance Criteria for Concentrations of PCBs in QC

	1668A				1668C		
Congener	Congener No.	VER (%)	OPR (%)	IS Samples(%)	VER (%)	OPR (%)	IS Samples(%)
3,4,4',5-Tetra-CB	80	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4-Penta-CB	81	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',5-Penta-CB	82	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',5-Penta-CB	83	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',6-Penta-CB 2,2',3,5,5'-Penta-CB	84/92	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,4'-Penta-CB 2,3,4,5,6-Penta-CB	85/116	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,5-Penta-CB	86	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,5'-Penta-CB 2,3,4',5,6-Penta-CB 2',3,4,5,6'-Penta-CB	87/117/125	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,6-Penta-CB 2,2',3,4',6-Penta-CB	88/91	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,6'-Penta-CB	89	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4',5-Penta-CB 2,2',4,5,5'-Penta-CB	90/101	70-130	50-150	NA	75-125	60-135	NA
2,2',3,5,6-Penta-CB	93	70-130	50-150	NA	75-125	60-135	NA
2,2',3,5,6'-Penta-CB	94	70-130	50-150	NA	75-125	60-135	NA
2,2',3,5',6-Penta-CB 2,2',3',4,6-Penta-CB 2,2',4,5,6'-Penta-CB	95/98/102	70-130	50-150	NA	75-125	60-135	NA
2,2',3,6,6'-Penta-CB	96	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4',5-Penta-CB	97	70-130	50-150	NA	75-125	60-135	NA
2,2',4,4',5-Penta-CB	99	70-130	50-150	NA	75-125	60-135	NA
2,2',4,4',6-Penta-CB	100	70-130	50-150	NA	75-125	60-135	NA
2,2',4,5',6-Penta-CB	103	70-130	50-150	NA	75-125	60-135	NA
2,2',4,4,6'-Penta-CB	104	70-130	50-150	NA	75-125	60-135	NA
2,3,3',4,4'-Penta-CB	105	70-130	50-150	NA	75-125	60-135	NA
2,3',4,4',5-Penta-CB 2,3,3',4,5-Penta-CB	118/106	70-130	50-150	NA	75-125	60-135	NA
2,3,3',4',5-Penta-CB 2,3,3',4,6-Penta-CB	107/109	70-130	50-150	NA	75-125	60-135	NA
2,3,3',4,5'-Penta-CB 2,3,3',5,6-Penta-CB	108/112	70-130	50-150	NA	75-125	60-135	NA
2,3,3',4',6-Penta-CB	110	70-130	50-150	NA	75-125	60-135	NA
2,3,3',5,5'-Penta-CB 2,3,4,4',6-Penta-CB	111/115	70-130	50-150	NA	75-125	60-135	NA
2,3,3',5',6-Penta-CB	113	70-130	50-150	NA	75-125	60-135	NA
2,3,4,4',5-Penta-CB	114	70-130	50-150	NA	75-125	60-135	NA
2,3',4,4',6-Penta-CB	119	70-130	50-150	NA	75-125	60-135	NA

Table 7
Acceptance Criteria for Concentrations of PCBs in QC

	1668A				1668C		
Congener	Congener No.	VER (%)	OPR (%)	IS Samples(%)	VER (%)	OPR (%)	IS Samples(%)
2,3',4,5,5'-Penta-CB	120	70-130	50-150	NA	75-125	60-135	NA
2,3',4,5',6-Penta-CB	121	70-130	50-150	NA	75-125	60-135	NA
2,3,3',4',5'-Penta-CB	122	70-130	50-150	NA	75-125	60-135	NA
2,3',4,4',5'-Penta-CB	123	70-130	50-150	NA	75-125	60-135	NA
2,3',4',5,5'-Penta-CB	124	70-130	50-150	NA	75-125	60-135	NA
3,3'4,4',5-Penta-CB	126	70-130	50-150	NA	75-125	60-135	NA
3,3',4,5,5'-Penta-CB	127	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4,4'-Hexa-CB 2,3,3',4',5,5'-Hexa-CB	128/162	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4,5-Hexa-CB	129	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4,5'-Hexa-CB	130	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4,6'-Hexa-CB 2,3,3',4,5',6-Hexa-CB	132/161	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4,6-Hexa-CB 2,2',3,3',5,5'Hexa-CB	131/133	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',5,6-Hexa-CB 2,2',3,4,5,6'-Hexa-CB	134/143	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',5,6'-Hexa-CB	135	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',6,6'-Hexa-CB	136	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,4',5-Hexa-CB	137	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,4',5'-Hexa-CB 2,3,3',4',5,6-Hexa-CB 2,3,3',4',5',6-Hexa-CB	138/163/164	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,4',6-Hexa-CB 2,2',3,4',5',6-Hexa-CB	139/149	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,4',6'-Hexa-CB	140	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,5,5'-Hexa-CB	141	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,5,6-Hexa-CB	142	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,5',6-Hexa-CB	144	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,6,6'-Hexa-CB	145	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4',5,5'-Hexa-CB 2,3,3',5,5',6-Hexa-CB	146/165	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4',5,6-Hexa-CB	147	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4',5,6'-Hexa-CB	148	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4',6,6'-Hexa-CB	150	70-130	50-150	NA	75-125	60-135	NA
2,2',3,5,5',6-Hexa-CB	151	70-130	50-150	NA	75-125	60-135	NA
2,2',3,5,6,6'-Hexa-CB	152	70-130	50-150	NA	75-125	60-135	NA
2,2',4,4',5,5'-Hexa-CB	153	70-130	50-150	NA	75-125	60-135	NA
2,2',4,4',5',6-Hexa-CB	154	70-130	50-150	NA	75-125	60-135	NA
2,2',4,4',6,6'-Hexa-CB	155	70-130	50-150	NA	75-125	60-135	NA

Table 7
Acceptance Criteria for Concentrations of PCBs in QC

	1668A				1668C		
Congener	Congener No.	VER (%)	OPR (%)	IS Samples(%)	VER (%)	OPR (%)	IS Samples(%)
2,3,3’,4,4’,5-Hexa-CB	156	70-130	50-150	NA	75-125	60-135	NA
2,3,3’,4,4’,5’-Hexa-CB	157	70-130	50-150	NA	75-125	60-135	NA
2,3,3',4,4',6-Hexa-CB 2,3,3',4,5,6-Hexa-CB	158/160	70-130	50-150	NA	75-125	60-135	NA
2,3,3’,4,5,5’-Hexa-CB	159	70-130	50-150	NA	75-125	60-135	NA
2,3,4,4’,5,6-Hexa-CB	166	70-130	50-150	NA	75-125	60-135	NA
2,3’,4,4’,5,5’-Hexa-CB	167	70-130	50-150	NA	75-125	60-135	NA
2,3’,4,4’,5’,6-Hexa-CB	168	70-130	50-150	NA	75-125	60-135	NA
3,3’,4,4’,5,5’-Hexa-CB	169	70-130	50-150	NA	75-125	60-135	NA
2,2’,3,3’,4,4’,5-Hepta-CB	170	70-130	50-150	NA	75-125	60-135	NA
2,2’,3,3’,4,4’,6-Hepta-CB	171	70-130	50-150	NA	75-125	60-135	NA
2,2’,3,3’,4,5,5’-Hepta-CB	172	70-130	50-150	NA	75-125	60-135	NA
2,2’,3,3’,4,5,6-Hepta-CB	173	70-130	50-150	NA	75-125	60-135	NA
2,2’,3,3’,4,5,6’-Hepta-CB	174	70-130	50-150	NA	75-125	60-135	NA
2,2’,3,3’,4,5’,6-Hepta-CB	175	70-130	50-150	NA	75-125	60-135	NA
2,2’,3,3’4,6,6’-Hepta-CB	176	70-130	50-150	NA	75-125	60-135	NA
2,2’,3,3’,4’,5,6-Hepta-CB	177	70-130	50-150	NA	75-125	60-135	NA
2,2’,3,3’,5,5’,6-Hepta-CB	178	70-130	50-150	NA	75-125	60-135	NA
2,2’,3,3’,5,6,6’-Hepta-CB	179	70-130	50-150	NA	75-125	60-135	NA
2,2’,3,4,4’,5,5’-Hepta-CB	180	70-130	50-150	NA	75-125	60-135	NA
2,2’,3,4,4’,5,6-Hepta-CB	181	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,4',5,6'-Hepta-CB 2,2',3,4',5,5',6-Hepta-CB	182/187	70-130	50-150	NA	75-125	60-135	NA
2,2’,3,4,4’,5’,6-Hepta-CB	183	70-130	50-150	NA	75-125	60-135	NA
2,2’,3,4,4’,6,6’-Hepta-CB	184	70-130	50-150	NA	75-125	60-135	NA
2,2’,3,4,5,5’,6-Hepta-CB	185	70-130	50-150	NA	75-125	60-135	NA
2,2’,3,4,5,6,6’-Hepta-CB	186	70-130	50-150	NA	75-125	60-135	NA
2,2’,3,4’,5,6,6’-Hepta-CB	188	70-130	50-150	NA	75-125	60-135	NA
2,3,3’,4,4’,5,5’-Hepta-CB	189	70-130	50-150	NA	75-125	60-135	NA
2,3,3’,4,4’,5,6-Hepta-CB	190	70-130	50-150	NA	75-125	60-135	NA
2,3,3’,4,4’,5’,6-Hepta-CB	191	70-130	50-150	NA	75-125	60-135	NA
2,3,3’,4,5,5’,6-Hepta-CB	192	70-130	50-150	NA	75-125	60-135	NA
2,3,3’,4’,5,5’,6-Hepta-CB	193	70-130	50-150	NA	75-125	60-135	NA
2,2’,3,3’,4,4’,5,5’-OctaCB	194	70-130	50-150	NA	75-125	60-135	NA
2,2’,3,3’,4,4’,5,6-OctaCB	195	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4,4',5,6'-OctaCB 2,2',3,4,4',5,5',6-OctaCB	196/203	70-130	50-150	NA	75-125	60-135	NA
2,2’,3,3’,4,4’,6,6’-OctaCB	197	70-130	50-150	NA	75-125	60-135	NA
2,2’,3,3’,4,5,5’,6-OctaCB	198	70-130	50-150	NA	75-125	60-135	NA
2,2’,3,3’,4,5,5’,6’-OctaCB	199	70-130	50-150	NA	75-125	60-135	NA
2,2’,3,3’,4,5,6,6’-OctaCB	200	70-130	50-150	NA	75-125	60-135	NA

Table 7
Acceptance Criteria for Concentrations of PCBs in QC

	1668A				1668C		
Congener	**Congener No.**	**VER (%)**	**OPR (%)**	**IS Samples(%)**	**VER (%)**	**OPR (%)**	**IS Samples(%)**
2,2',3,3',4,5',6,6'-OctaCB	201	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',5,5',6,6'-OctaCB	202	70-130	50-150	NA	75-125	60-135	NA
2,2',3,4,4',5,6,6'-OctaCB	204	70-130	50-150	NA	75-125	60-135	NA
2,3,3',4,4',5,5',6-OctaCB	205	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4,4',5,5',6-NoCB	206	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4,4',5,6,6'-NoCB	207	70-130	50-150	NA	75-125	60-135	NA
2,2',3,3',4,5,5',6,6'-NoCB	208	70-130	50-150	NA	75-125	60-135	NA
De-CB	209	70-130	50-150	NA	75-125	60-135	NA
Internal Standards							
^{13}C-2-MonoCB	1	50-150	15-140	15-150	50-145	15-145	5-145
^{13}C-4-MonoCB	3	50-150	15-140	15-150	50-145	15-145	5-145
^{13}C-2,2'-DiCB	4	50-150	30-140	25-150	50-145	15-145	5-145
^{13}C-2,5-DiCB	9	50-150	30-140	25-150	50-145	15-145	5-145
^{13}C-3,3'-DiCB	11	50-150	30-140	25-150	50-145	15-145	5-145
^{13}C-2,2',6-Tri-CB	19	50-150	30-140	25-150	50-145	15-145	5-145
^{13}C-2,4,4'-Tri-CB	28	50-150	30-140	25-150	50-145	15-145	5-145
^{13}C-2,4',6-Tri-CB	32	50-150	30-140	25-150	50-145	15-145	5-145
^{13}C-3,4,4'-Tri-CB	37	50-150	30-140	25-150	50-145	15-145	5-145
^{13}C-2,2',4,4'-Tetra-CB	47	50-150	30-140	25-150	50-145	15-145	5-145
^{13}C-2,2',5,5'-Tetra-CB	52	50-150	30-140	25-150	50-145	15-145	5-145
^{13}C-2,2',6,6'-Tetra-CB	54	50-150	30-140	25-150	50-145	15-145	5-145
^{13}C-2,3',4',5-Tetra-CB	70	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-3,3',4,4'-Tetra-CB	77	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-3,4,4',5-Tetra-CB	80	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-3,3',4,4'-Tetra-CB	81	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,2',3,5',6-Penta-CB	95	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,2',3,4',5-Penta-CB	97	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,2',4,5,5'-Penta-CB	101	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,2',4,6,6'-Penta-CB	104	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,3,3',4,4'-Penta-CB	105	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,3,4,4',5-Penta-CB	114	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,3',4,4',5-Penta-CB	118	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2',3,4,4',5-Penta-CB	123	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-3,3',4,4',5-Penta-CB	126	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-3,3',4,5,5'-Penta-CB	127	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,2',3,4,4',5'-Hexa-CB	138	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,2',3,4,5,5'-Hexa-CB	141	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,2',4,4',5,5'-Hexa-CB	153	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,2',4,4',6,6'-Hexa-CB	155	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,3,3',4,4',5-Hexa-CB	156	50-150	30-140	25-150	50-145	40-145	10-145

Table 7
Acceptance Criteria for Concentrations of PCBs in QC

	1668A				1668C		
Congener	**Congener No.**	**VER (%)**	**OPR (%)**	**IS Samples(%)**	**VER (%)**	**OPR (%)**	**IS Samples(%)**
^{13}C-2,3,3',4,4',5'-Hexa-CB	157	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,3,3',4,5,5'-Hexa-CB	159	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,3',4,4',5,5'-Hexa-CB	167	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-3,3',4,4',5,5'-Hexa-CB	169	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,2',3,3',4,4',5-Hepta-CB	170	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,2',3,4,4',5,5'-Hepta-CB	180	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,2',3,4',5,6,6'-Hepta-CB	188	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,3,3',4,4',5,5'-Hepta-CB	189	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,2',3,3',4,4',5,5'-OctaCB	194	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,2',3,3',5,5',6,6'-OctaCB	202	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,2',3,3',4,4',5,5',6-NonaCB	206	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-2,2',3,3',4,5,5',6,6'-NonaCB	208	50-150	30-140	25-150	50-145	40-145	10-145
^{13}C-Deca-CB	209	50-150	30-140	25-150	50-145	40-145	10-145
Cleanup Recovery Standards							
^{13}C-3,3',4,5'-Tetra-CB[1]	79	60-130	40-125	30-135	75-125	40-145	10-145
^{13}C-2,2'3,3'5,5'6-Hepta-CB[1]	178	60-130	40-125	30-135	75-125	40-145	10-145

Table 8

PCB Natives and Corresponding Labeled Compounds

Congener	Labeled	Recovery Standard
PCB-1	^{13}C-PCB-1	^{13}C-PCB-15
PCB-2	^{13}C-PCB-3	^{13}C-PCB-15
PCB-3	^{13}C-PCB-3	^{13}C-PCB-15
PCB-4/10	^{13}C-PCB-4	^{13}C-PCB-15
PCB-7/9	^{13}C-PCB-9	^{13}C-PCB-15
PCB-6	^{13}C-PCB-9	^{13}C-PCB-15
PCB-5/8	^{13}C-PCB-9	^{13}C-PCB-15
PCB-14	^{13}C-PCB-11	^{13}C-PCB-15
PCB-11	^{13}C-PCB-11	^{13}C-PCB-15
PCB-12/13	^{13}C-PCB-11	^{13}C-PCB-15
PCB-15	^{13}C-PCB-11	^{13}C-PCB-15
PCB-19	^{13}C-PCB-19	^{13}C-PCB-15
PCB-30	^{13}C-PCB-19	^{13}C-PCB-15
PCB-18	^{13}C-PCB-32	^{13}C-PCB-15
PCB-17	^{13}C-PCB-32	^{13}C-PCB-15
PCB-24/27	^{13}C-PCB-32	^{13}C-PCB-15
PCB-16/32	^{13}C-PCB-32	^{13}C-PCB-15
PCB-34	^{13}C-PCB-28	^{13}C-PCB-31
PCB-23	^{13}C-PCB-28	^{13}C-PCB-31
PCB-29	^{13}C-PCB-28	^{13}C-PCB-31
PCB-26	^{13}C-PCB-28	^{13}C-PCB-31
PCB-25	^{13}C-PCB-28	^{13}C-PCB-31
PCB-31	^{13}C-PCB-28	^{13}C-PCB-31
PCB-28	^{13}C-PCB-28	^{13}C-PCB-31
PCB-20/21/33	^{13}C-PCB-28	^{13}C-PCB-31
PCB-22	^{13}C-PCB-28	^{13}C-PCB-31
PCB-36	^{13}C-PCB-37	^{13}C-PCB-31
PCB-39	^{13}C-PCB-37	^{13}C-PCB-31
PCB-38	^{13}C-PCB-37	^{13}C-PCB-31
PCB-35	^{13}C-PCB-37	^{13}C-PCB-31
PCB-37	^{13}C-PCB-37	^{13}C-PCB-31
PCB-54	^{13}C-PCB-54	^{13}C-PCB-60
PCB-50	^{13}C-PCB-54	^{13}C-PCB-60
PCB-53	^{13}C-PCB-52	^{13}C-PCB-60
PCB-51	^{13}C-PCB-52	^{13}C-PCB-60
PCB-45	^{13}C-PCB-52	^{13}C-PCB-60
PCB-46	^{13}C-PCB-52	^{13}C-PCB-60
PCB-52/69	^{13}C-PCB-52	^{13}C-PCB-60
PCB-73	^{13}C-PCB-52	^{13}C-PCB-60
PCB-43/49	^{13}C-PCB-52	^{13}C-PCB-60

Table 8

PCB Natives and Corresponding Labeled Compounds

Congener	Labeled	Recovery Standard
PCB-47	^{13}C-PCB-47	^{13}C-PCB-60
PCB-48/75	^{13}C-PCB-47	^{13}C-PCB-60
PCB-65	^{13}C-PCB-47	^{13}C-PCB-60
PCB-62	^{13}C-PCB-47	^{13}C-PCB-60
PCB-44	^{13}C-PCB-47	^{13}C-PCB-60
PCB-42/59	^{13}C-PCB-47	^{13}C-PCB-60
PCB-41/64/71/72	^{13}C-PCB-47	^{13}C-PCB-60
PCB-68	^{13}C-PCB-47	^{13}C-PCB-60
PCB-40	^{13}C-PCB-47	^{13}C-PCB-60
PCB-57	^{13}C-PCB-70	^{13}C-PCB-60
PCB-67	^{13}C-PCB-70	^{13}C-PCB-60
PCB-58	^{13}C-PCB-70	^{13}C-PCB-60
PCB-63	^{13}C-PCB-70	^{13}C-PCB-60
PCB-74	^{13}C-PCB-70	^{13}C-PCB-60
PCB-61/70	^{13}C-PCB-70	^{13}C-PCB-60
PCB-76/66	^{13}C-PCB-70	^{13}C-PCB-60
PCB-80	^{13}C-PCB-80	^{13}C-PCB-60
PCB-55	^{13}C-PCB-80	^{13}C-PCB-60
PCB-56/60	^{13}C-PCB-80	^{13}C-PCB-60
PCB-79	^{13}C-PCB-80	^{13}C-PCB-60
PCB-78	^{13}C-PCB-81	^{13}C-PCB-60
PCB-81	^{13}C-PCB-81	^{13}C-PCB-60
PCB-77	^{13}C-PCB-77	^{13}C-PCB-60
PCB-104	^{13}C-PCB-104	^{13}C-PCB-111
PCB-96	^{13}C-PCB-104	^{13}C-PCB-111
PCB-103	^{13}C-PCB-104	^{13}C-PCB-111
PCB-100	^{13}C-PCB-104	^{13}C-PCB-111
PCB-94	^{13}C-PCB-95	^{13}C-PCB-111
PCB-95/98/102	^{13}C-PCB-95	^{13}C-PCB-111
PCB-93	^{13}C-PCB-95	^{13}C-PCB-111
PCB-88/91	^{13}C-PCB-95	^{13}C-PCB-111
PCB-121	^{13}C-PCB-95	^{13}C-PCB-111
PCB-84/92	^{13}C-PCB-101	^{13}C-PCB-111
PCB-89	^{13}C-PCB-101	^{13}C-PCB-111
PCB-90/101	^{13}C-PCB-101	^{13}C-PCB-111
PCB-113	^{13}C-PCB-101	^{13}C-PCB-111
PCB-99	^{13}C-PCB-101	^{13}C-PCB-111
PCB-119	^{13}C-PCB-97	^{13}C-PCB-111
PCB-108/112	^{13}C-PCB-97	^{13}C-PCB-111
PCB-83	^{13}C-PCB-97	^{13}C-PCB-111
PCB-97	^{13}C-PCB-97	^{13}C-PCB-111

Table 8

PCB Natives and Corresponding Labeled Compounds

Congener	Labeled	Recovery Standard
PCB-86	^{13}C-PCB-97	^{13}C-PCB-111
PCB-87/117/125	^{13}C-PCB-97	^{13}C-PCB-111
PCB-111/115	^{13}C-PCB-97	^{13}C-PCB-111
PCB-85/116	^{13}C-PCB-97	^{13}C-PCB-111
PCB-120	^{13}C-PCB-97	^{13}C-PCB-111
PCB-110	^{13}C-PCB-97	^{13}C-PCB-111
PCB-82	^{13}C-PCB-123	^{13}C-PCB-111
PCB-124	^{13}C-PCB-123	^{13}C-PCB-111
PCB-107/109	^{13}C-PCB-123	^{13}C-PCB-111
PCB-123	^{13}C-PCB-123	^{13}C-PCB-111
PCB-106/118	^{13}C-PCB-118	^{13}C-PCB-111
PCB-114	^{13}C-PCB-114	^{13}C-PCB-128
PCB-122	^{13}C-PCB-114	^{13}C-PCB-128
PCB-105	^{13}C-PCB-105	^{13}C-PCB-128
PCB-127	^{13}C-PCB-127	^{13}C-PCB-128
PCB-126	^{13}C-PCB-126	^{13}C-PCB-128
PCB-155	^{13}C-PCB-155	^{13}C-PCB-111
PCB-150	^{13}C-PCB-155	^{13}C-PCB-111
PCB-152	^{13}C-PCB-155	^{13}C-PCB-111
PCB-145	^{13}C-PCB-155	^{13}C-PCB-111
PCB-136	^{13}C-PCB-155	^{13}C-PCB-111
PCB-148	^{13}C-PCB-155	^{13}C-PCB-111
PCB-154	^{13}C-PCB-155	^{13}C-PCB-111
PCB-151	^{13}C-PCB-155	^{13}C-PCB-111
PCB-135	^{13}C-PCB-155	^{13}C-PCB-111
PCB-144	^{13}C-PCB-155	^{13}C-PCB-111
PCB-147	^{13}C-PCB-155	^{13}C-PCB-111
PCB-139/149	^{13}C-PCB-155	^{13}C-PCB-111
PCB-140	^{13}C-PCB-155	^{13}C-PCB-111
PCB-134/143	^{13}C-PCB-153	^{13}C-PCB-128
PCB-131/133	^{13}C-PCB-153	^{13}C-PCB-128
PCB-142	^{13}C-PCB-153	^{13}C-PCB-128
PCB-146/165	^{13}C-PCB-153	^{13}C-PCB-128
PCB-132/161	^{13}C-PCB-153	^{13}C-PCB-128
PCB-153	^{13}C-PCB-153	^{13}C-PCB-128
PCB-168	^{13}C-PCB-153	^{13}C-PCB-128
PCB-141	^{13}C-PCB-141	^{13}C-PCB-128
PCB-137	^{13}C-PCB-141	^{13}C-PCB-128
PCB-130	^{13}C-PCB-141	^{13}C-PCB-128
PCB-138/163/164	^{13}C-PCB-138	^{13}C-PCB-128
PCB-158/160	^{13}C-PCB-138	^{13}C-PCB-128

Table 8

PCB Natives and Corresponding Labeled Compounds

Congener	Labeled	Recovery Standard
PCB-129	^{13}C-PCB-138	^{13}C-PCB-128
PCB-166	^{13}C-PCB-159	^{13}C-PCB-128
PCB-159	^{13}C-PCB-159	^{13}C-PCB-128
PCB-128/162	^{13}C-PCB-159	^{13}C-PCB-128
PCB-167	^{13}C-PCB-167	^{13}C-PCB-128
PCB-156	^{13}C-PCB-156	^{13}C-PCB-128
PCB-157	^{13}C-PCB-157	^{13}C-PCB-128
PCB-169	^{13}C-PCB-169	^{13}C-PCB-128
PCB-188	^{13}C-PCB-188	^{13}C-PCB-128
PCB-184	^{13}C-PCB-188	^{13}C-PCB-128
PCB-179	^{13}C-PCB-188	^{13}C-PCB-128
PCB-176	^{13}C-PCB-188	^{13}C-PCB-128
PCB-186	^{13}C-PCB-188	^{13}C-PCB-128
PCB-178	^{13}C-PCB-188	^{13}C-PCB-128
PCB-175	^{13}C-PCB-188	^{13}C-PCB-128
PCB-182/187	^{13}C-PCB-188	^{13}C-PCB-128
PCB-183	^{13}C-PCB-188	^{13}C-PCB-128
PCB-185	^{13}C-PCB-180	^{13}C-PCB-128
PCB-174	^{13}C-PCB-180	^{13}C-PCB-128
PCB-181	^{13}C-PCB-180	^{13}C-PCB-128
PCB-177	^{13}C-PCB-180	^{13}C-PCB-128
PCB-171	^{13}C-PCB-180	^{13}C-PCB-128
PCB-173	^{13}C-PCB-180	^{13}C-PCB-128
PCB-172	^{13}C-PCB-180	^{13}C-PCB-128
PCB-192	^{13}C-PCB-180	^{13}C-PCB-128
PCB-180	^{13}C-PCB-180	^{13}C-PCB-128
PCB-193	^{13}C-PCB-180	^{13}C-PCB-128
PCB-191	^{13}C-PCB-180	^{13}C-PCB-128
PCB-170	^{13}C-PCB-170	^{13}C-PCB-128
PCB-190	^{13}C-PCB-170	^{13}C-PCB-128
PCB-189	^{13}C-PCB-189	^{13}C-PCB-128
PCB-202	^{13}C-PCB-202	^{13}C-PCB-128
PCB-201	^{13}C-PCB-202	^{13}C-PCB-128
PCB-204	^{13}C-PCB-202	^{13}C-PCB-128
PCB-197	^{13}C-PCB-202	^{13}C-PCB-128
PCB-200	^{13}C-PCB-202	^{13}C-PCB-128
PCB-198	^{13}C-PCB-202	^{13}C-PCB-128
PCB-199	^{13}C-PCB-202	^{13}C-PCB-128
PCB-196/203	^{13}C-PCB-202	^{13}C-PCB-128
PCB-195	^{13}C-PCB-194	^{13}C-PCB-205
PCB-194	^{13}C-PCB-194	^{13}C-PCB-205

Table 8

PCB Natives and Corresponding Labeled Compounds

Congener	Labeled	Recovery Standard
PCB-205	^{13}C-PCB-194	^{13}C-PCB-205
PCB-208	^{13}C-PCB-208	^{13}C-PCB-205
PCB-207	^{13}C-PCB-208	^{13}C-PCB-205
PCB-206	^{13}C-PCB-206	^{13}C-PCB-205
PCB-209	^{13}C-PCB-209	^{13}C-PCB-205
^{13}C-PCB-15 (RS)		
^{13}C-PCB-31 (RS)		
^{13}C-PCB-60 (RS)		
^{13}C-PCB-111 (RS)		
^{13}C-PCB-128 (RS)		
^{13}C-PCB-205 (RS)		
	^{13}C-PCB-79 (CRS)	^{13}C-PCB-60
	^{13}C-PCB-178 (CRS)	^{13}C-PCB-128
	^{13}C-PCB-79 (PS)	^{13}C-PCB-81
	^{13}C-PCB-178 (PS)	^{13}C-PCB-180

Table 9. - Quality Control Requirements For PCBs by Modified Method 1668A/C					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Instrument Tuning	Beginning and end of each shift	Using a PFK molecular leak, tune the instrument to meet minimum required resolving power of 10,000 at or close to m/z 304.9824. An appropriate lock mass will be monitored for each descriptor and shall not vary by more than ± 20% throughout the respective retention time window. For the measurement of PCBs, the exact m/z's to be monitored in each descriptor are listed in Table 6.	If the lock mass varies by more than +20%, the data must be evaluated to determine whether the batch shall be re-extracted or the data are qualified appropriately. Dilute and re-analyze	Not applicable	Because of the extensive mass range covered in each function, it may not be possible to maintain 10,000 resolutions throughout the mass range. Therefore, resolution must be 8,000 throughout the mass range and must be 10,000 in the center of the mass range for each function.
Initial Calibration (ICAL)	An initial calibration is repeated whenever a new set of spiking calibration standards are created or whenever the continuing calibration falls outside the acceptance criteria	The signal to noise ratio (s/n) must exceed 10:1 for all ions monitored (except Di-CBs are at or above 2.5:1). The ion abundance ratio measurements must be within the theoretical ratio limits (See Table 3). Calibration by Isotope Dilution: Isotope dilution calibration is used for the native PCBs for which labeled compounds are available. If the relative response for any compound is less than 20% RSD over the 6-point calibration range, an averaged relative response is used for that compound.	If this criteria are not achieved, a new initial calibration curve must be re-injected or prepared	Not applicable	

Table 9. - Quality Control Requirements For PCBs by Modified Method 1668A/C					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Initial Calibration (ICAL)		Calibration by Internal Standard: Natives for which a labeled compound is not available will use internal standard calibration. The average Relative Response for that congener will be used and %RSD must be < 30%. All initial instrument calibrations are verified with a standard solution from a second manufacturer or lot. The in-house limits of 60-140% have been developed as the acceptance criteria based upon second source standard history.			
Initial Calibration Verification (ICV)/Continuing Calibration	Prior to running samples/with every batch	Each compound must be within the verification limits established in Table 7. The ion ratios must be within the theoretical ratio limits (Table 3). The signal to noise ratio (s/n) must exceed 10:1 for all ions monitored. The absolute retention times of the internal standards shall be within ±15 seconds of the retention times obtained during calibration. The relative retention times of the peak for a native and labeled PCB should be within	If the stated criteria are not met for any compound, correct the problem and repeat the calibration verification, or recalibrate and document return to control.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Refer to laboratory flagging criteria and project requirements.	

Table 9. - Quality Control Requirements For PCBs by Modified Method 1668A/C					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Initial Calibration Verification (ICV)/Continuing Calibration		0.5% of the retention time windows established from the initial calibration curve			
Method Blank (MB)	With every analytical batch or 20 samples, whichever is less, per matrix type Analyze the method or solvent blank immediately after analysis of the OPR to demonstrate freedom from contamination	≤ minimum level or one-third of the regulatory compliance limit, whichever is greater; Or ≤ minimum level for each congener	If the amount found is greater than the minimum level or one-third the regulatory compliance limit, whichever is greater; or if any potentially interfering compound is found in the blank at or above the minimum level for each congener, the data must be evaluated to determine whether the batch shall be re-extracted or the data is qualified appropriately.	Refer to flagging criteria and project requirements.	
Matrix Spike (MS)	By client request or to fulfill state agency requirements	The relative percent difference between MS/MSD samples should be ≤25%	If the criteria are not met, the data must be evaluated to determine whether the samples shall be re-extracted or the data are qualified appropriately.	Not applicable	Per client request only Projects performed pursuant to the guidelines established by the DOD QSM shall contain an associated Matrix Spike per preparatory batch. A Matrix Spike Duplicate or Laboratory Duplicate shall also be analyzed per preparatory batch for these projects
Matrix Spike Duplicate (MSD) or Matrix	Per client request only	The relative percent difference between MS/MSD	If the criteria are not met, the data must be evaluated to determine	Not applicable	For DoD projects: Refer to QSM limits.

Table 9. - Quality Control Requirements For PCBs by Modified Method 1668A/C					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Duplicate (MD)		samples should be ≤25% Criteria for qualitative and quantitative determinations of chromatographic peaks must be met.	whether the samples shall be re-extracted or the data are qualified appropriately.		MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD or sample and MD).
Internal Standards	Added to every sample	For acceptance criteria, see Table 7 The absolute retention times of the internal standards shall be within ±15 seconds of the retention times obtained during calibration		Not applicable	
Ongoing Precision and Recovery (OPR)	with every analytical batch or 20 samples (whichever is less) per matrix type	For each native and labeled compound, compare the concentration with the limits for ongoing accuracy in Table 7.	If IS is low and native compounds are in, assess the data and determine whether to re-extract or narrate. If native compounds are out, re-extract.	Not applicable	

GLOSSARY

Symbols

°C	Celsius
L	microliter
m	micrometer
<	less than
>	greater than
%	percent

Alphabetical Abbreviations

cm	centimeter
g	gram
L	liter
M	molecular ion
m	meter
mg	milligram
ml	milliliter
mm	millimeter
m/z	mass-to-charge ration
N	normal
pg	picogram
ppb	part-per-billion
ppm	part-per-million
ppq	part-per-quadrillion
ppt	part-per-trillion
v/v	volume per unit volume
w/v	weight per unit volume

Definitions and Acronyms

Analyte – a PCB tested for by this method. The analytes are listed in Table 1.

Calibration Standard – a solution prepared from a secondary standard and/or stock solutions used to calibrate the response of the instrument.

Calibration Verification Standard – the mid-point calibration standard (CS-3) that is used to verify calibration.

CB – chlorinated biphenyl congener.

CS-1, CS-2, CS-3, CS-4, CS-5 – See calibration standards and Table 2.

HRMS – High resolution mass spectrometry.

Internal Standard – an internal standard is a labeled PCB which is added to all field samples, blanks and other quality control samples before extraction. It is also included in the calibration solutions. Internal standards are used to measure the concentration of the analyte and surrogate compounds.

IPR – initial precision and recovery, four aliquots of a reference matrix spiked with the analytes of interest and labeled compounds and analyzed to establish the ability of the laboratory to generated acceptable precision and recovery.

Isotope Dilution – a means of determining a naturally occurring (native) compound by reference to the same compound in which one or more atoms has been isotopically enriched.

Matrix Spike (MS) – a sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte is concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on the methods recovery efficiency.

Method Blank (MB) – an aliquot of reagent water that is treated exactly as a sample including exposure to all glassware, equipment, solvents, regents, internal standards, and surrogates that

are used with samples. The method blank is used to determine if analytes or interferences are present in the laboratory environment, the reagents, or the apparatus.

Minimum Level - The level at which the entire analytical system must give a recognizable signal and acceptable calibration point for the analyte. It is equivalent to the concentration of the lowest calibration standard, assuming that all Method-specified sample weights, volumes, and cleanup procedures have been employed.

OPR – ongoing precision and recovery standard, a method blank spiked with known quantities of analytes. The OPR is analyzed exactly like a sample. Its purpose is to assure that the results produced by the laboratory remain within the limits specified in this method for precision and recovery.

PCB – polychlorinated biphenyl

PFK – perfluorokerosene, a mixture of compounds used to calibrate the exact m/z scale in the HRMS.

Practical Quantitation Limit (PQL) – The PQL is a limit for each compound at or below which data must not be reported.

Reagent Water – water demonstrated to be free from the analytes of interest and potentially interfering substances at the method detection limit for the analyte.

Recovery Standard – A recovery standard is a labeled compound, which is added to the extracts of all samples, blanks, and QC samples before analysis. It is also included in the calibration solutions. The response of the internal standards relative to the recovery standard is used to estimate the recovery of the internal standards. The internal standard recovery is an indicator of the overall performance of the analysis.

Relative Response Factor – The relative response factor is the response of the mass spectrometer to a known amount of an analyte or labeled compound relative to a known amount of an internal standard or another labeled compound.

Signal-to-Noise Ratio (S/N) – the height of the signal as measured from the mean (average) of the noise to the peak maximum divided by the width of the noise.

Stock Solution – a solution containing an analyte that is prepared using a reference material traceable to a source that will attest to the purity and authenticity of the reference material.

Description of Amendment
Requirements for extraction and analysis of samples from the State of Wisconsin: All samples from the State of Wisconsin shall be analyzed only on instruments with a valid LOD study for that parameter, matrix, and method, i.e., DoD instruments. All samples from the State of Wisconsin shall be extracted with a measurement of precision, such as an MS/MSD, LCSD or sample duplicate, per batch and precision assessed. All reportable values for samples from the State of Wisconsin shall be within the range of calibration. Concentrations shall not be "E" qualified. PCB Congeners detected in the Method Blank must be less than the higher of the following criteria: 1. One-third of the lowest standard in the Initial Calibration. 2. One-twentieth of the regulatory limit. 3. One-tenth of the sample concentration.

ATTACHMENT 5

EPA Method 1668C
Aqueous Method Detection Limit/Matrix Reporting Limit

Analyte	MDL		MRL	Units
PCB-1	1.30		5.00	pg/L
PCB-2	1.57		5.00	pg/L
PCB-3	1.36		5.00	pg/L
PCB-4/10	2.27		10.0	pg/L
PCB-5/8	2.05		10.0	pg/L
PCB-6	1.71		5.00	pg/L
PCB-7/9	2.31		10.0	pg/L
PCB-11	6.05		5.00	pg/L
PCB-12/13	3.65		10.0	pg/L
PCB-14	1.44		5.00	pg/L
PCB-15	1.12		5.00	pg/L
PCB-16/32	1.90		10.0	pg/L
PCB-17	1.48		5.00	pg/L
PCB-18	1.97		5.00	pg/L
PCB-19	1.55		5.00	pg/L
PCB-20/21/33	7.79		15.0	pg/L
PCB-22	2.78		5.00	pg/L
PCB-23	3.00		5.00	pg/L
PCB-24/27	3.15		10.0	pg/L
PCB-25	2.64		5.00	pg/L
PCB-26	2.45		5.00	pg/L
PCB-28	3.36		5.00	pg/L
PCB-29	1.92		5.00	pg/L
PCB-30	1.66		5.00	pg/L
PCB-31	2.42		5.00	pg/L
PCB-34	1.08		5.00	pg/L
PCB-35	2.14		5.00	pg/L
PCB-36	2.02		5.00	pg/L
PCB-37	1.71		5.00	pg/L
PCB-38	1.97		5.00	pg/L
PCB-39	2.21		5.00	pg/L
PCB-40	2.65		5.00	pg/L
PCB-41/64/71/72	7.11		20.0	pg/L
PCB-42/59	3.10		10.0	pg/L
PCB-43/49	4.28		10.0	pg/L
PCB-44	2.98		5.00	pg/L
PCB-45	1.82		5.00	pg/L
PCB-46	1.79		5.00	pg/L
PCB-47	2.52		5.00	pg/L
PCB-48/75	4.61		10.0	pg/L
PCB-50	1.91		5.00	pg/L
PCB-51	1.36		5.00	pg/L
PCB-52/69	3.88		10.0	pg/L

EPA Method 1668C
Aqueous Method Detection Limit/Matrix Reporting Limit

Analyte	MDL		MRL	Units
PCB-53	2.19		5.00	pg/L
PCB-54	2.17		5.00	pg/L
PCB-55	2.10		5.00	pg/L
PCB-56/60	4.08		10.0	pg/L
PCB-57	2.31		5.00	pg/L
PCB-58	3.01		5.00	pg/L
PCB-61/70	3.78		10.0	pg/L
PCB-62	1.98		5.00	pg/L
PCB-63	1.52		5.00	pg/L
PCB-65	2.61		5.00	pg/L
PCB-66/76	3.95		10.0	pg/L
PCB-67	2.69		5.00	pg/L
PCB-68	2.49		5.00	pg/L
PCB-73	1.69		5.00	pg/L
PCB-74	2.32		5.00	pg/L
PCB-77	2.76		5.00	pg/L
PCB-78	1.95		5.00	pg/L
PCB-79	2.34		5.00	pg/L
PCB-80	2.47		5.00	pg/L
PCB-81	2.76		5.00	pg/L
PCB-82	0.878		5.00	pg/L
PCB-83	1.16		5.00	pg/L
PCB-84/92	3.81		10.0	pg/L
PCB-85/116	2.25		10.0	pg/L
PCB-86	3.08		5.00	pg/L
PCB-87/117/125	6.11		15.0	pg/L
PCB-88/91	6.75		5.00	pg/L
PCB-89	1.55		5.00	pg/L
PCB-90/101	4.58		10.0	pg/L
PCB-93	1.86		5.00	pg/L
PCB-94	2.17		5.00	pg/L
PCB-95/98/102	4.86		15.0	pg/L
PCB-96	1.13		5.00	pg/L
PCB-97	3.04		5.00	pg/L
PCB-99	2.20		5.00	pg/L
PCB-100	1.54		5.00	pg/L
PCB-103	1.72		5.00	pg/L
PCB-104	1.42		5.00	pg/L
PCB-105	1.50		5.00	pg/L
PCB-106/118	3.19		10.0	pg/L
PCB-107/109	2.07		10.0	pg/L
PCB-108/112	3.49		10.0	pg/L
PCB-110	0.800		5.00	pg/L

EPA Method 1668C
Aqueous Method Detection Limit/Matrix Reporting Limit

Analyte	MDL		MRL	Units
PCB-111/115	3.35		10.0	pg/L
PCB-113	3.03		5.00	pg/L
PCB-114	2.84		5.00	pg/L
PCB-119	1.57		5.00	pg/L
PCB-120	1.82		5.00	pg/L
PCB-121	4.01		5.00	pg/L
PCB-122	3.56		5.00	pg/L
PCB-123	1.93		5.00	pg/L
PCB-124	2.40		5.00	pg/L
PCB-126	0.793		5.00	pg/L
PCB-127	1.02		5.00	pg/L
PCB-128/162	2.81		10.0	pg/L
PCB-129	2.41		5.00	pg/L
PCB-130	0.764		5.00	pg/L
PCB-131/133	1.65		5.00	pg/L
PCB-132/161	3.39		10.0	pg/L
PCB-142	2.56		10.0	pg/L
PCB-134/143	2.82		10.0	pg/L
PCB-135	2.19		5.00	pg/L
PCB-136	1.63		5.00	pg/L
PCB-137	1.59		5.00	pg/L
PCB-138/163/164	4.00		15.0	pg/L
PCB-139/149	3.61		10.0	pg/L
PCB-140	2.14		5.00	pg/L
PCB-141	1.54		5.00	pg/L
PCB-144	4.16		5.00	pg/L
PCB-145	3.32		5.00	pg/L
PCB-146/165	3.07		10.0	pg/L
PCB-147	4.01		5.00	pg/L
PCB-148	4.82		5.00	pg/L
PCB-150	1.07		5.00	pg/L
PCB-151	2.42		5.00	pg/L
PCB-152	2.69		5.00	pg/L
PCB-153	1.82		5.00	pg/L
PCB-154	4.28		5.00	pg/L
PCB-155	2.44		5.00	pg/L
PCB-156	1.76		5.00	pg/L
PCB-157	1.14		5.00	pg/L
PCB-158/160	2.43		10.0	pg/L
PCB-159	1.75		5.00	pg/L
PCB-166	1.36		5.00	pg/L
PCB-167	1.70		5.00	pg/L
PCB-168	1.04		5.00	pg/L

EPA Method 1668C
Aqueous Method Detection Limit/Matrix Reporting Limit

Analyte	MDL		MRL	Units
PCB-169	2.27		5.00	pg/L
PCB-170	3.33		5.00	pg/L
PCB-171	1.55		5.00	pg/L
PCB-172	2.68		5.00	pg/L
PCB-173	2.52		5.00	pg/L
PCB-174	1.94		5.00	pg/L
PCB-175	2.18		5.00	pg/L
PCB-176	2.57		5.00	pg/L
PCB-177	2.38		5.00	pg/L
PCB-178	2.60		5.00	pg/L
PCB-179	2.60		5.00	pg/L
PCB-180	1.52		5.00	pg/L
PCB-181	1.85		5.00	pg/L
PCB-182/187	3.48		10.0	pg/L
PCB-183	1.79		5.00	pg/L
PCB-184	1.40		5.00	pg/L
PCB-185	1.02		5.00	pg/L
PCB-186	2.69		5.00	pg/L
PCB-188	1.98		5.00	pg/L
PCB-189	1.49		5.00	pg/L
PCB-190	2.11		5.00	pg/L
PCB-191	0.950		5.00	pg/L
PCB-192	1.79		5.00	pg/L
PCB-193	1.42		5.00	pg/L
PCB-194	1.33		5.00	pg/L
PCB-195	1.48		5.00	pg/L
PCB-196/203	5.56		10.0	pg/L
PCB-197	2.45		5.00	pg/L
PCB-198	4.50		5.00	pg/L
PCB-199	3.98		5.00	pg/L
PCB-200	3.32		5.00	pg/L
PCB-201	1.65		5.00	pg/L
PCB-202	1.24		5.00	pg/L
PCB-204	2.47		5.00	pg/L
PCB-205	1.82		5.00	pg/L
PCB-206	2.18		5.00	pg/L
PCB-207	1.32		5.00	pg/L
PCB-208	0.887		5.00	pg/L
PCB-209	2.28		5.00	pg/L

GENERAL ELECTRIC COMPANY
HUDSON RIVER PHASE 2 REMEDIAL ACTION MONITORING PROGRAM
2017 CORRECTIVE ACTION MEMORANDUM No. 15

Date: August 15, 2017, February 6, 2018 (revised)
Organization Name: Environmental Standards, Inc.
Initiator's Name and Title: Meg Michell

Problem Description: According to Section 3 of the Phase 2 Remedial Action Monitoring Quality Assurance Project Plan (Phase 2 RAM QAPP), fish samples are to be analyzed for PCBs (Aroclor PCBs by SW-846 8082A and congener-specific PCBs by modified Green Bay Method [mGBM]) and lipids in accordance with the procedures and analytical methods specified in Attachment A. According to Section 3 of Attachment A, these analyses were to be performed according to the Pace Analytical Services, LLC Schenectady, NY (Pace Schenectady) standard operating procedures (SOPs) contained in Appendices A3-1 through A3-4. The Pace Schenectady laboratory facility closed operations at the end of 2016. In addition, the mGBM is not offered at any other commercial laboratory facility. The purpose of this Corrective Action Memorandum (CAM) is to document the analytical procedures that will be used for fish samples collected in 2017 through the beginning of the long-term operation, maintenance, and monitoring (OM&M) program with the exception of the analysis for congener-specific PCBs. As stated in Section 3.5.1 of the Phase 2 RAM QAPP, PCB congener analysis will be performed on 5% of the total number of fish samples, during every other sampling event that is conducted at a given sampling location. Since PCB congener analysis was performed on 5% of the fish collected in 2016, PCB congener analysis will not be required in 2017.

February 6, 2018 CAM15 Update: EPA provided comments on November 8, 2017 to GE to CAM15. GE responded to EPA's comments on December 22, 2017 and incorporated changes in the Standard Operating Procedures referenced below to address EPA's comments. EPA approved CAM15 on January 26, 2018. EPA's approval letter describes the agreed upon analysis by the Pace Analytical Services, LLC Green Bay, WI (Pace Green Bay) of select 2016 fish tissue samples for Aroclor PCBs by GEHR 8082 and an appropriate PCB congener method. Details of this study will be submitted to EPA in a separate plan. The approval letter also indicates GE will incorporate an appropriate PCB congener method for 5% of the 2018 fish and will include use of a reference material. This CAM15 update includes the updated and revised Standard Operating Procedures.

Reported To: Bob Gibson, GE

Corrective Action: Pace Green Bay will perform the preparation and analysis of fish tissue samples for Aroclor PCBs and lipids. Pace Green Bay will be performing these analyses using procedures similar to that of Pace Schenectady, as detailed below.

Fish tissue samples will first be prepared and homogenized following the methods outlined in Attachment 1 (Pace Green Bay SOP S-GB-L-009-REV.01), which replaces Pace Schenectady SOP S-NY-O-333-rev.05 (Appendix A3-3 of Attachment A). Both the Green Bay and Schenectady SOPs are based on the guidance provided in Draft Procedures for Collection and Preparation of Aquatic Biota for Contaminant Analysis (NYSDEC 2002) and the NYSDEC Hale Creek Field Station SOP PrepLab4 (5-28-2014), Prep Lab Standard Operating Procedure

(NYSDEC 2014) as specified in Attachment A. Pace Green Bay SOP S-GB-L-009-REV.01 is directly based on Pace Schenectady SOP S-NY-O-333-rev.05; however, the following changes were incorporated into Pace Green Bay SOP S-GB-L-009-REV.01.

1. Removed references to biota material other than fish.
2. Added the US Berkel meat grinder to the process equipment list.
3. Added using the US Berkel meat grinder to grind whole body fish or larger fillets.
4. Updated the equipment list to match Pace Green Bay laboratory equipment.
5. Added methanol to the reagent list for utensil and counter cleaning.
6. Removed Hexane from reagent list.
7. Added an alternative process to remove the skin from catfish and bullheads.
8. Removed pre-chilling the food process before tissue is ground because it can cause the blades to freeze up and/or break off.

Extraction and cleanup of homogenized fish tissue and lipids analysis will be performed as detailed in the SOP provided as Attachment 2 (Pace Green Bay SOP S-GB-O-068-REV.01), which replaces Pace Schenectady SOP S-NY-O-017-rev.14 (Appendix A3-4 of Attachment A) for extraction and lipids analysis and Pace Schenectady SOPs for sulfuric acid clean-up (Appendix A2-5 of Attachment A, SW-846 Method 3665A, SOP S-NY-O-338-rev.00) and Florisil clean-up (Appendix A2-6 of Attachment A, SW-846 Method 3620C, SOP S-NY-O-340-rev.00). Sulfur clean-up (Appendix A2-4 of Attachment A, SW-846 Method 3660B, Pace SOP S-NY-O-337-rev.01) will no longer be performed. Pace Green Bay SOP S-GB-O-068-REV.01 is directly based on the aforementioned Pace Schenectady SOPs; however, the following changes were incorporated into Pace Green Bay SOP S-GB-O-068-REV.01.

1. Updated equipment list to match Pace Green Bay procedures.
2. Soxtherm extraction by SW-846 Method 3541 replaced Soxhlet extraction by SW-846 Method 3540C.
3. Removed 1:1 magnesium sulfate/sodium sulfate and replaced it with sodium sulfate only.
4. Changed the surrogate spike concentrations from 0.5/5.0ug/mL TCMX/DCB to 2.0ug/mL for both TCMX and DCB.
5. Final volume of extract was changed from 25mL to 5mL to keep the reporting limit (RL) the same.
6. Removed the copper cleanup step for the removal of Elemental Sulfur. Sulfur interference is uncommon in fish tissue samples.
7. Changed the amount of extract for lipid determination from 10mLs of a 25mL extract to 1mL of a 5mL extract.
8. Moved the screening of extracts to the analytical SOP.

Fish tissue extracts will be analyzed for Aroclor PCBs following SW-846 Method 8082A as detailed in the laboratory SOP provided as Attachment 3 (Pace Green Bay SOP S-GB-O-067-REV.01), which replaces Pace Schenectady SOP S-NY-O-314-rev.03a (Appendix A3-1 of Attachment A). Pace Green Bay SOP S-GB-O-067-REV.01 is directly based on Pace Schenectady SOP S-NY-O-314-rev.03a; however, the following changes were incorporated into Pace Green Bay SOP S-GB-O-067-REV.01. Please note that a number of these changes were made because Pace Green Bay SOP S-GB-O-067-REV.01 will be a project-specific SOP whereas Pace Schenectady SOP S-NY-O-314-rev.03a was a general laboratory SOP.

1. Switched calibration to Average Response Factor option from inverse weighted linear calibration curve.
2. Switched quantitation to be based on average of peak by peak quantitation vs area sum of peaks.
3. Removed Aroclors 1262 and 1268 from compound list per Phase 2 RAM QAPP analyte list.
4. Remove reference to soil, water, sediment, sludge, oil, waste solvent since only fish tissue will be performed by Pace Green Bay.
5. Updated the reporting limit table to reflect the calibration and final volume changes.
6. Removed Elemental Sulfur from the interferences list. Sulfur interference not found in biota.
7. Updated Definitions.
8. Changed reference of “Continuing Calibration Check Standard” (CCCS) to “Continuing Calibration Verification Standard” (CCV) – same source as the calibration standard where the CCCS was previously a second source.
9. Added “Initial Calibration Verification Standard” (ICV) – second source from the calibration standard
10. The ICV will be analyzed immediately following the initial calibration to confirm the calibration is made properly. The instrument calibration will then be confirmed by the analysis of CCVs.
11. Instruments windows will be opened and closed with one of the rotated Aroclors.
12. Updated the instrument to include Agilent 6890 and injector.
13. Added Chemstation and Target software to the Equipment list.
14. Removed reference to the secondary column (ZB-5).
15. Updated equipment list to match Pace Green Bay’s equipment list.
16. Updated Stock standard tables to Green Bay sources.
17. Updated standard preparation tables to match Green Bay’s calibration levels.
18. Updated to Pace Green Bay’s calibration level concentrations.
19. Removed surrogated instrument blank after CCVs. The instrument blanks never showed carryover from the CCV occurred.
20. Removed reference to Empower processing system.
21. Removed the section about Dual Column confirmation as results are only reported from the ZB-1 phase column
22. Updated the Quality control tables.
    a. Updated LCS, MS and MSD limits to 70-130% per the Phase 2 RAM QAPP.
    b. Updated that the LCS, MS and MSD will be spiked with Aroclor 1242 as specified in Phase 2 RAM QAPP.
    c. Updated surrogate acceptance limits to 60-140% to match Phase 2 RAM QAPP
23. Removed A-note/P-note qualitative identification comments.

The laboratory RLs and measurement performance criteria for precision, accuracy/bias, representativeness, comparability, completeness, and sensitivity will be the same as used by Pace Schenectady and summarized in Attachment A Tables A1-2, A3-1a, and A3-1c. The laboratory is in the process of conducting MDL studies for Aroclor PCBs following the attached

procedures. The MDLs will be provided to the EPA upon completion of the MDL studies. New York does not provide certification of laboratories for PCB and lipids analysis in fish tissue.

Reviewed and Implemented By: David Blye (Environmental Standards)

Copy to: GE Program Manager: Bob Gibson
QA Program Manager: David Blye (Environmental Standards)

ATTACHMENT 1

Pace Analytical Services, LLC – Green Bay WI
1241Bellevue Street
Green Bay, WI 54302

Phone: 920 469 2436
Fax: 920 469 8827

STANDARD OPERATING PROCEDURE

TISSUE PREPARATION/HOMOGENIZATION OF FISH FOR GE HUDSON RIVER MONITORING PROGRAMS

Reference Methods: N/A

SOP Number:	S-GB-L-009-Rev.01
Effective Date:	Upon Final Signature
Supersedes:	S-NY-O-333-Rev.05 (Appendix A3-3 of RAM QAPP Attachment A)

APPROVALS

Nils K. Melberg, General Manager — Date: 01/31/18

Kate E. Verbeten, Quality Manager — Date: 1/30/18

Chris Haase, Department Manager — Date: 1/30/18

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

TABLE OF CONTENTS

SECTION **PAGE**

1. Purpose/Identification of Method

1.1. This is the Standard Operating Procedure (SOP) for tissue preparation, processing, and homogenization prior to extraction/digestion and analysis for the GE Hudson River Monitoring Programs.

2. Summary of Method

2.1. **Fish**: Samples are weighed, measured, and sex determined if possible. The fish may be processed as a whole body for ecological assessment or as fillets following the method developed by the New York State Department of Environmental Conservation (NYSDEC). Following the NYSDEC method, the left fillet will be taken except in the following circumstances: (a) Fish less than approximately 6 inches long and rainbow smelt will be prepared by cutting the head off from behind the pectoral fin and eviscerating the fish (b) if there is insufficient mass (less than 50 g), the right fillet will be taken in addition to the left and homogenized together as described in Section 12.1.7.5. If compositing of multiple fish is required, the fish samples identified as requiring filleting that are also to be composited will be filleted and/or skinned prior to homogenization, while the entire whole bodies of fish collected for ecological assessment (i.e., forage fish) will be composited and homogenized together. The carcass of the fish (after removal of the fillet) may be maintained for separate homogenization and analysis if requested, until the client requests disposal of the carcass.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP are applicable to all personnel involved in the preparation and homogenization of fish for GE Hudson River Monitoring Programs.

3.2. **Parameters**: See applicable analytical SOPs for analyte lists.

4. Applicable Matrices

4.1. This method is applicable to the preparation and homogenization of fish (whole body and fillets).

5. Limits of Detection and Quantitation

5.1. Not applicable to this SOP.

6. Interferences

6.1. Samples being tested for organics generally must be processed with metal, Teflon, PTFE and/or glass utensils. Wood tongue depressors may also be used to transfer samples. The use of plastics may cause interferences with the analysis of samples.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Sample collection is not applicable to the Pace laboratory operation.

7.2. Please see the Pace SOP (S-GB-C-010 current version) that describes the responsibilities of sample custody including all proper documentation, verification, and tracking procedures following Chain of Custody

(COC) protocols, sample receipt procedures, and Internal COC procedures for sample tracking include the use of sample tracking logbooks.

7.3. Each sample is received from the field sampler in clean aluminum foil inside a labeled plastic re-sealable storage bag.

7.4. Fish arrive whole bodied. Once received the sample must be filleted when required then ground and homogenized so that it may be analyzed. The remaining carcass is retained once the fillets are cut out until the client requests disposal of the carcass.

7.5. If samples are not shipped frozen, they will be stored in freezers at Pace Analytical Services upon arrival, and until processing. The samples must remain frozen and maintained at $\leq -10^{o}C$ unless being tested. The extraction hold time for organics is one year from sample collection to extraction when frozen at $\leq -10^{o}C$, and 40 days from extraction to analysis. NOTE: It is the processing technician's responsibility to put away all fish carcass and fish homogenates into frozen storage at the end of the day when processing is complete.

7.6. Tissue samples: As guidance, a minimum of 50g of sample must be collected for organic analysis in a glass jar with Teflon or PTFE lined screw cap. The amount of sample needed, will depend upon the project management plan such as reporting limits and the need for MS/MSD and/or duplicate analyses. Extra sample must be collected, if possible, to allow the laboratory adequate sample volume in case of re-extract and re-analysis is needed. Large whole individual fillets may be wrapped in plastic or aluminum foil depending upon the requested analyses.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

8.2. Biota- the flora or fauna of a region.

8.3. Caudal- directed toward or situated in or near the tail or posterior part of the body.

8.4. Composite- combining the typical or essential characteristics of individuals, making up a group.

8.5. Fillet- to cut an edible portion of fish including the ribcage and belly flap from the left side of the fish, unless there is insufficient mass, in which case the fillet will also include the right side The left side is defined as the side facing the technician when the fish's head is on the technician's left and the belly is proximal to the technician.

8.6. Head- the upper or anterior division of the animal body that contains the brain, the chief sense organs, and the mouth.

8.7. Homogenize- to reduce particles so that they are uniformly small, of uniform color and consistency, with no free fluid, and evenly distributed. The portion of sample that has been homogenized is referred to as the homogenate throughout this SOP.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Cutting board-made of either glass or polyethylene.

9.2. US Berkel meat grinder model E-222.

9.3. Food processor with titanium cutting blade (small), or blender with stainless steel blades (large):

9.3.1. Robot Coupe R2 Ultra Stainless commercial food processor

9.3.2. Waring Commercial ProPrep Chopper Grinder Model Number: WCG75 with designated grinding bowl/blade and chopping bowl/blade

9.4. Knives: ceramic, stainless steel, or titanium. (See Section 6 for interferences and/or contamination associated with different material knives and blades).

9.4.1. Stainless Steel Boning knives.

9.4.2. Stainless Steel Chopping knives.

9.4.3. Stainless Steel fillet knives.

9.4.4. URI Eagle Ceramic Knife.

9.4.5. Scissors

9.4.6. Serrated general purpose stainless steel knife

9.5. Analytical Balance: Sargent-Welch 400DR capable of weighing 15000±0.1grams (or equivalent) used to determine sample mass.

9.6. Bench liner material (Lab Mat) and scissors.

9.7. Aluminum foil.

9.8. Plastic wrap or wax paper.

9.9. Teflon-coated spatula.

9.10. Stainless steel Spoon/spatula – Fisher S50822/14-375-20

9.11. Teflon or stainless steel tweezers and dissection scissors.

9.12. PVC or Latex gloves

9.13. Ruler.

9.14. Mallet.

9.15. Stainless steel or plastic strainer.

9.16. Salad Spinner.

9.17. Pre-cleaned glass sample jars with Teflon or PTFE-lined caps.

9.18. Kim wipes.

9.19. Nylon bristled brushes for cleaning.

9.20. Digital camera.

9.21. Wood tongue depressor.

9.22. Pyrex glass tray.

10. Reagents and Standards

10.1. Deionized (DI) water- Deionized (DI) water or reagent water is ASTM Type II laboratory reagent grade water or better (Type I)..

10.2. Methanol – reagent grade Fisher A412-20 .

10.3. 10% Bleach solution - Add 100mL of commercial bleach to 500mL of reagent water and dilute to 1 liter in an appropriate beaker or flask.

10.4. Alconox - cleaning solution.

11. Calibration and Standardization

11.1. Not applicable to this SOP.

12. Procedure

12.1. Fish Sample Preparation

12.1.1. Cover lab benches with clean bench liner material as needed before processing each fish.

12.1.2. Weighing whole fish

12.1.2.1. Determine the wet weight for each individual fish or composite sample using a top loaded balance. The balance must pass the daily calibration verification before usage. A series of NIST-certified weights that encompass the total weight of samples (whole body fish, fillets, and homogenates) being weighed are used to verify the calibration. Measure and record the weight in biota homogenization logbook using the following procedure:

12.1.2.2. Cover scale with aluminum foil (for fluid retention)

12.1.2.3. Zero the balance (tare)

12.1.2.4. Place the fish or composite sample on the top loading balance and place bar coded sample ID label in front of the scale.

12.1.2.5. Using the digital camera take a picture of the fish or composite sample and the ID label. Include the weight from the balance in the picture. Retain the picture for project records.

12.1.2.6. Record the weight of the fish or composite sample in the biota homogenization logbook.

12.1.3. If the sample is an individual fish to be filleted, proceed to Section 12.1.4.

12.1.3.1. If the sample is a whole body sample (individual or composite), proceed to Section 12.1.7.6.

12.1.4. Preparation before filleting

12.1.4.1. If the fish is to be filleted with skin on, proceed to Section 12.1.5 (see Section 12.1.10 for species-specific preparation guidance)

12.1.4.2. If the fish is to be filleted with skin off, proceed to Section 12.1.6 (see Section 12.1.10 for species-specific preparation guidance)

12.1.5. Scaling the fish:

12.1.5.1. Lay the fish (right side down) on a clean cutting board

12.1.5.2. Scrape the left side of the fish from tail to head using a manual scaler or the electric automated descaler with ceramic claws to remove the scales.

12.1.5.3. Proceed to Section 12.1.7.

12.1.6. Skinning the fish

12.1.6.1. Place the fish (right side down) on a clean cutting board to skin left side of fish. To skin the fish, loosen the skin behind the gill, cover and pull the skin off toward the tail with a catfish skinning tool, cutting lightly along the inside of the skin. Slowly separate the skin from the muscle tissue of the body of the fillet. Alternatively, the skin can be removed from the fillet by laying the removed fillet,

skin side down on the cutting board, then sliding the knife down the fillet between the skin and the muscle of the fish until the skin is separated (See 12.1.7 for fillet removal).

12.1.6.2. For skin-off samples (e.g., catfish or bullheads), rinse the outside of the fish with DI water and pat dry with paper towel to remove mucilaginous secretions. Proceed to Section 12.1.7.

12.1.7. Filleting the fish and preparing samples for homogenization

12.1.7.1. Lay the fish in the cutting board on its right side.

12.1.7.2. Make a diagonal cut from the base of the cranium following just behind the gill to the ventral side just behind the pectoral fin. Remove the flesh, ribcage and belly flap from the left side of the fish by cutting from the cranium along the spine and dorsal rays to the caudal fin. (Be careful not to cut into and contaminate the fillet with internal organs.) For specimens that have large ribcage bones, poultry scissors may be helpful in cutting through the ribcage.

12.1.7.3. Identify the sex of the specimen (if possible) and record the sex in the biota homogenization prep logbook. If sex determination is not possible, record as indeterminate.

12.1.7.4. Note if the internal organs were ruptured during freezing or if inadvertent puncture of the internal organs occurred during the filleting process in the sample processing records in biota homogenization prep logbook. Rinse the fillet(s) tissue with DI water.

12.1.7.5. If the sample is a fillet obtained from Sections 12.1.7.1-12.1.7.4, place the fillet on the balance covered with aluminum foil and weigh. If there is insufficient mass (i.e., less than 40 g) from the left fillet, take the right fillet starting at Section 12.1.3 and reversing the sides. Add the right fillet to the left fillet on the balance. Record the weight of the fillet(s) in the biota homogenization prep logbook and denote if fillets were combined. Store the leftover carcass (including skin for skin off fillets) in the freezer in its original field collection bag.

12.1.7.6. Estimate the total number of containers needed for the processed fish based on weight of the tissue to be homogenized (fillet or whole body fish) and the appropriate sized jar. Generate container labels from LIMS. Affix label(s) to containers(s). Place the containers on the analytical balance and zero the balance (tare). If original estimate of labels is insufficient, re-print label set to accommodate jars used, noting that jars are designated on the label as x of y (*e.g.*, "jar 1 of 2," "jar 2 of 2").

12.1.7.7. Chop the fillet(s) or whole body sample into smaller pieces (chunks of approximately one inch size) on a clean cutting board.

12.1.8. Homogenization of fillet and whole body fish (individual or composite) samples

12.1.8.1. If proceeding directly from Section 12.1.7.7, transfer fillet or whole body sample to the appropriately sized food processor or grinder. Transfer any excess fluids from the aluminum foil (from Section 12.1.7.5 for fillet samples or from Section 12.1.2 for whole body samples) and cutting board from Section 12.1.7.7 with sample. In addition, scrape chopped remnants and fluids from cutting board into the food processor using a clean, stainless steel flat edged butcher knife or wood tongue depressor.

12.1.8.2. If the sample is frozen (from Section 12.1.7.9), allow the chopped fillet(s) or whole body sample to partially thaw. Transfer chopped fillet to the appropriately sized food processor or grinder using a clean, stainless steel flat edged butcher knife or wood tongue depressor. Retain all fluids in container as part of the sample to be homogenized.

12.1.8.3. Homogenize whole fish bodies (individual or composite samples) or fillets by placing them into the appropriately sized food processor fitted with the appropriate blades or a commercial sized meat grinder. For total fillet weight exceeding 200 grams, the Robot Coupe R2 processor is used. For very large fillets that will not fit in the Robot Coupe R2 bowl, the fillet is processed in multiple batches (typically, up to 3 batches). For smaller fish, either the Waring commercial "Grind" or "Chop" bowls are used based on efficacy. Process the sample until it appears to be fully and consistently homogenous. After approximately 30 seconds of grinding, stop food processor and mix and push tissue back toward food processor blade using the wood tongue depressor. Continue to grind, stop, and mix the sample until the sample is homogeneous (i.e., until a uniform paste-like consistency and color is observed with no phase separation). Typically, three 30 second grinds are used; however, some samples may require many short bursts to achieve desired homogeneity. Be sure to grind the sample sufficiently so that there are not whole sections of skin visually obvious in the sample. However, the food processor should not be ran until the point that it starts to warm the sample and the motor begins to heat excessively.

- For fillets that are too large to grind all at once, grind a subset of the chopped fillet from Section 12.1.8.2 in separate batches. As each batch is ground, place each batch in the same glass tray. When all chunks of the fillet have been ground, combine all of the ground tissue in the glass tray and thoroughly mix the tissue together to create a homogenized product.
- For large, whole fish or fillets over 200 grams, chop the fish into pieces small enough to be place into the grinder. Grind the entire fish 3 times, mixing the ground material between each grind.

12.1.8.4. The homogeneous nature of the sample is vitally important for reproducible results.

12.1.8.5. Transfer homogenized sample into tared jars from Section 12.1.7.6 using a clean stainless steel spoon/spatula or wood tongue depressor. When placing homogenized samples into multiple jars for storage, each jar should contain an amount of skin vs. tissue in the same approximate proportion to the overall sample volume. (This sample proportionality should also be used when weighting volume out for extraction/digestion.) Jars should only be filled to approximately ¾ of the jar volume to allow for further mixing/homogenization prior to extraction.

12.1.8.6. Once jars are filled, place all jars containing homogenate (with labels facing forward) on analytical balance (with bar coded sample ID label from Section 12.1.2.4 still in front of the balance) and weigh. Record weight of homogenate in biota homogenization prep logbook. Using the digital camera take a photograph of the jarred homogenate sample and the ID label. Include the weight from the balance in the picture. Retain the picture for project records.

- If additional jars are needed beyond the estimated number in Section 12.1.7.6, write the sample ID on the lid of the additional jar(s) needed and reprint container labels from LIMS. Affix label to new jar(s) and place on balance and tare. Transfer additional homogenate to the new jars and weigh. Add weight of additional homogenate in new jars to initial homogenate weight in biota homogenization prep logbook. Using the digital camera take a photograph of the additional jarred homogenate sample and the ID label. Include the weight from the balance in the picture. Retain the picture for project records. Make note in biota homogenization prep logbook that additional jar(s) were required and that multiple photographs were taken. Relabel original jars with newly printed labels so that the total number of jars is noted on all jars containing homogenate sample.

12.1.8.7. If the samples will not be extracted or digested immediately, the sample must be returned to the freezer until extraction/digestion.

12.1.8.8. All cutting boards, utensils and equipment must be washed between samples according to the procedures described in section 13.2.

12.1.9. Modifications to NYSDEC Standard Fillet Procedure

12.1.9.1. The following modifications of the standard fillet procedure are designed to account for variations in fish size or known preferred preparation.

12.1.9.2. Supplemental methods for human consumption

12.1.9.3. Some fish are too small to fillet by the above procedures. Fish less than approximately 6 inches long and rainbow smelt are prepared by cutting the head off from behind the pectoral fin and eviscerating the fish. Ensure that the belly flap is retained on the carcass to be analyzed. When this modification is used it should be noted when reporting analytical results. The flesh and ribcage are retained as the "fillet" sample.

12.1.9.4. Prepare American eel by removing the head, skin and viscera; do not attempt to fillet.

12.1.10. Species-specific fillet preparations

12.1.10.1. The following species are prepared as fillets with skin on

- largemouth bass
- smallmouth bass
- white perch
- yellow perch
- striped bass
- carp
- walleye

12.1.10.2. The following species are prepared as fillets with skin off:

- brown bullhead
- yellow bullhead
- white catfish
- channel catfish

13. Quality Control

13.1. Contamination Prevention:

13.1.1. If the purity of a reagent is in question, analyze for contamination.

13.1.2. Blades for filleting/dissection may need to be re-sharpened between every few samples as needed. Food processor blades should be replaced when they become dull. Spare processing blades are kept on hand.

13.2. The procedures described below are general cleaning and pre-processing procedures that are to be followed regardless of the type of tissue being processed. Samples are prioritized by the Laboratory Supervisor or Lab Manager based on hold time and client due date. All weights, measurements, and other project required observations are recorded in LIMS.

13.2.1. Wash all utensils, sample processors (blades, blade post, cup and lid) and cutting boards with an Alconox solution and a bristle brush. Rinse thoroughly with tap water, then with DI water.

13.2.2. Gloves must be worn when handling tissue samples. Latex gloves may be worn. All gloves must be talc or dust free.

13.2.3. Tissue samples should be partially thawed before starting, to the point where it becomes possible to make an incision in, or cut through, the flesh. When samples are completely thawed they become soft and difficult to cut or fillet. NOTE: If whole bodies are not being processed, and the tissue is partially frozen during dissection, there is less of a chance of puncturing the gut cavity and any internal organs. Inadvertent puncture of the internal organs may contaminate the part(s) of the animal that have been selected for analysis. Also, internal organs may rupture during freezing. If this is observed during dissection, it must be noted in the processing records. Note any morphological abnormalities on the processing records. Abnormalities may include growths or apparent tumors on internal organs, especially reproductive organs, discolored tissues, etc.

13.3. Hold times: The homogenized fish tissue can be held for 12 months from collection when frozen $\leq$ -10 degrees Celsius. The fish solvent extracts can be held for 40 days, or according to project specifications.

14. Data Analysis and Calculations

14.1. Not applicable to this SOP.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Not applicable to this SOP.

16. Corrective Actions for Out-of-Control Data

16.1. Not applicable to this SOP.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Not applicable to this SOP.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

19. Method Modifications

19.1. Not applicable to this SOP.

20. Instrument/Equipment Maintenance

20.1. Not applicable to this SOP.

21. Troubleshooting

21.1. Not applicable to this SOP.

22. Safety

22.1. The use of laboratory equipment and chemicals exposes the analyst to several potential hazards. Good laboratory techniques and safety practices shall be followed at all times. Approved PPE, which includes safety glasses and gloves, must be worn at all times in the lab. Lab coats are provided and must be worn. All Personal Protective Equipment (PPE) must be removed before leaving the laboratory area and before entering the employee lounge or eating area. Always wash your hands before leaving the laboratory.

22.2. All standards, reagents, and solvents shall be handled under a hood using the proper PPE. All flammable solvents must be kept in the flammable storage cabinet, and returned to the cabinet immediately after use. When transporting chemicals, make sure to use a secure transporting devise and/or secondary outer container.

22.3. The technician should have received in-house safety training and should know the location of the first aid equipment and the emergency spill/clean-up equipment before handling any apparatus or equipment.

22.4. Extreme caution must be taken when using or handling knives, de-scalers, and grinders to homogenize the biota samples.

22.5. Re-useable cotton mesh glove liners may be worn under latex or PVC gloves as an additional measure when using sharp tools or knives, or when dealing with samples that have sharp teeth, spines, fins, or thorns. The mesh lining can help prevent piercing of the skin in case a tool or sample slips, during dissection or other preparation steps.

22.6. Polychlorinated biphenyls should be treated with extreme caution; as a class of chemical compounds they possess both toxic and suspected carcinogenic properties.

22.7. All additional company safety practices shall be followed at all times as written in the Pace Analytical Health and Safety Plan.

23. Waste Management

23.1. Refer to the latest version of SOP S-GB-W-0001 for instructions on the disposal of waste generated during the procedures previously mentioned.

24. Pollution Prevention

24.1. The laboratory Chemical Hygiene Plan/Health and Safety Plan contains information on pollution prevention.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, “Quality Systems”- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, “Quality Systems”- most current version.

25.4. U.S.EPA SW-846 "Test Methods for Evaluating Solid Waste; Volume 1B Laboratory Manual Physical/Chemical Methods", Office of Solid Waste and Emergency Response, Third Edition, Final Update III, December 1996.

25.5. EPA/6OOlR-961027, Guidance for the Preparation of Standard Operating Procedures (SOPS) for Quality Related Documents, 1996.

25.6. US EPA 823-R-95-007, “Guidance for Assessing Chemical Contaminated Data for Use in Fish Advisories”, Volume 1: Fish Sampling and Analysis 2nd Edition, Office of Science and Technology, Office of Water, 1995.

25.7. US-EPA Fillet Procedure, 1991.

25.8. NYSDEC Draft Procedures for Collection and Preparation of Aquatic Biota for Contaminant Analysis, 2002.

25.9. NYSDEC Prep Lab SOP, Hale Creek Field Station, SOP PrepLab4 (5/28/2014).

25.10. Pace SOP S-GB-C-010 “Sample Management”

25.11. Pace SOP S-GB-W-001 “Waste Handling and Management”

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I: Anatomy of a Fish (Typical salmonid).

26.2. Attachment II: External Anatomy of a Striped Bass.

26.3. Attachment III: Typical Measurement Locations.

26.4. Attachment IV: Typical Measurements of Largemouth Bass.

26.5. Attachment V: Internal Anatomy.

26.6. Attachment VI: Kidneys and Testes.

27. Revisions

Document Number	Reason for Change	Date
S-GB-L-009-Rev.00	Used Pace – Schenectady Laboratory SOP S-NY-O-133 to create local SOP	14Jul2017
S-GB-L-009-Rev.00	Cover page: Updated Title to replace "Biota and Plant Material" to "Fish", updated Supersedes Section to: S-NY-O-333-Rev.05 (Appendix A3-3 of RAM QAPP Attachment A). Section 2.1: Clarified insufficient mass as <50g. Section 3.1: Replaced "biota and plant material" to "fish". Section 9.5: Changed from 300±0.01g to 15000±0.1g Section(s) 12.1.7.8. and 12.1.7.9.: Deleted section since laboratory will not stop mid-way through fish processing. Section(s) 12.1.8.1. and 12.1.8.2.: changed equipment from spoon to flat edged butcher knife.	29Nov2017

Attachment I: Anatomy of a Fish (Typical salmonid)

EXTERNAL ANATOMY

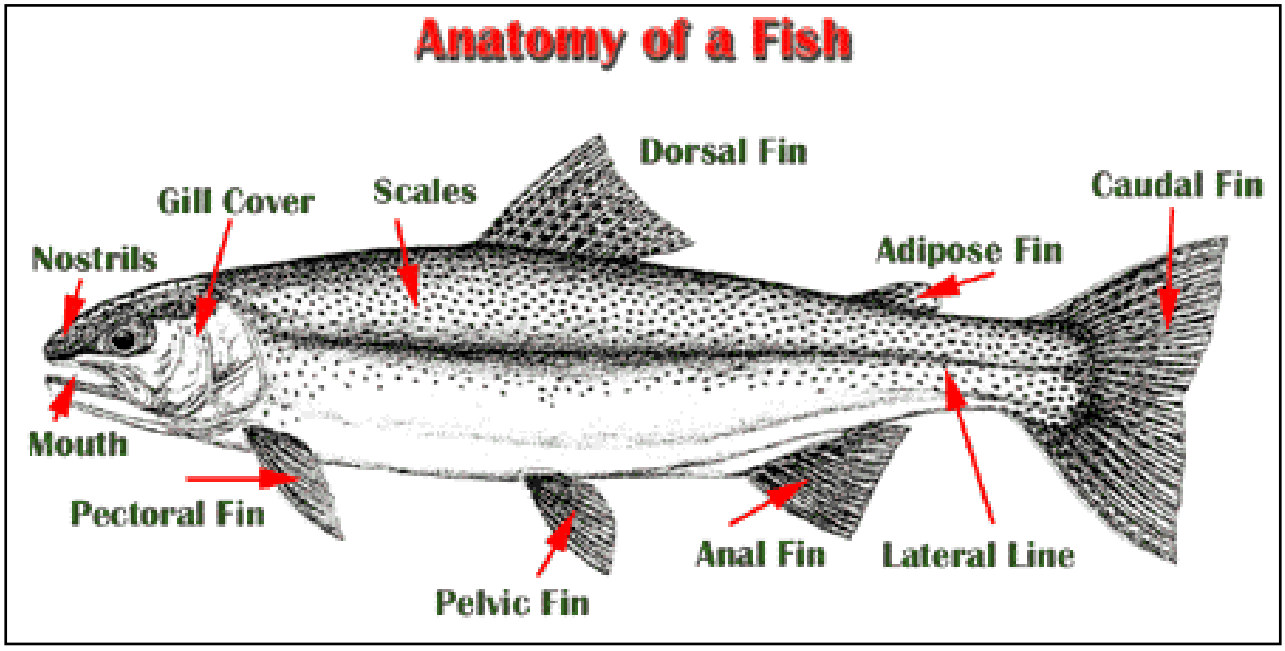
Remove one fish from the storage tank, place in dissecting pan. Make sure fish is euthanized prior to any dissection.

Locate all fins (Figures 1a and 1b):

Paired: pectoral (caudal to head, located ventrolaterally)
pelvic (cranial to anus, located ventrolaterally)

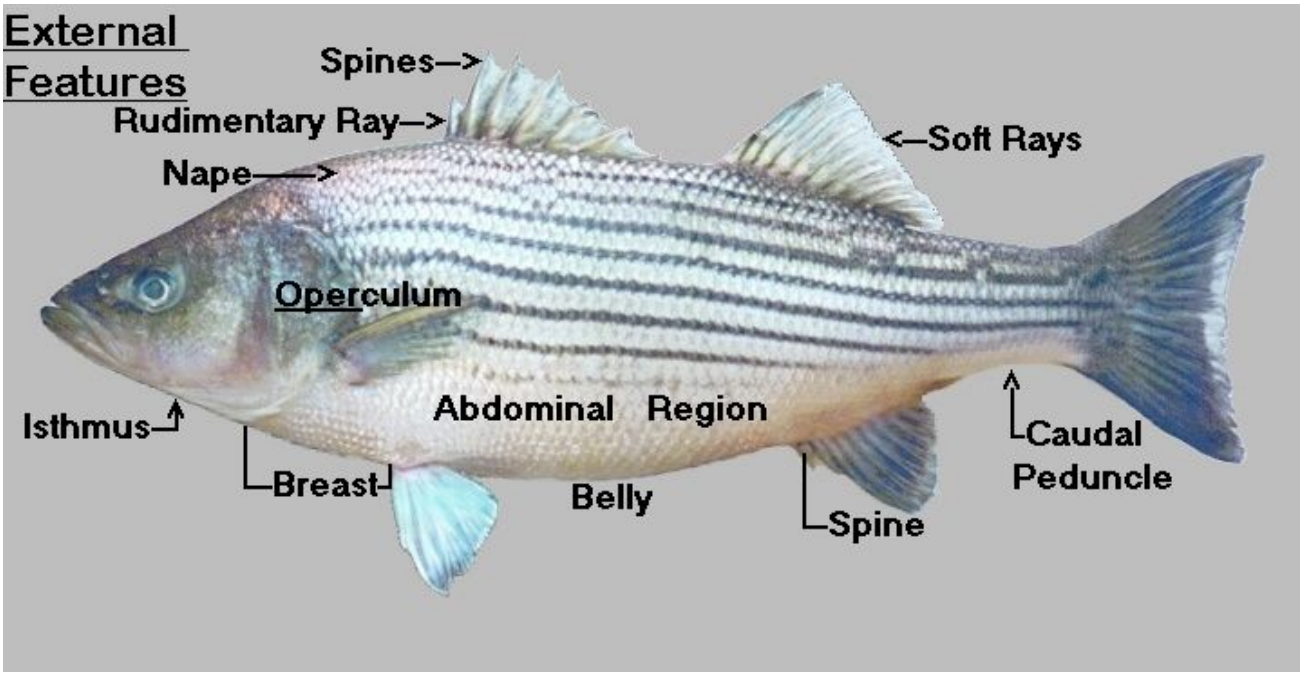
Single: dorsal (caudal to head on dorsal midline)
adipose (caudal to dorsal fin on dorsal midline; salmonids)

Anal: (Caudal to anus on ventral midline)

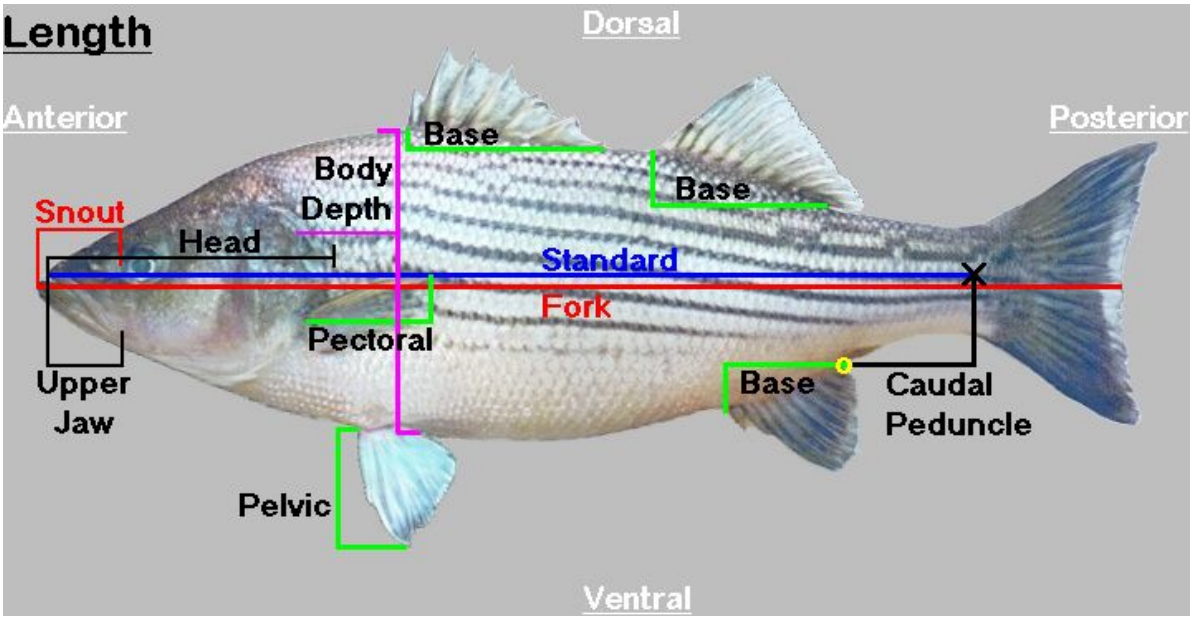


Attachment II: External Anatomy of a Striped Bass

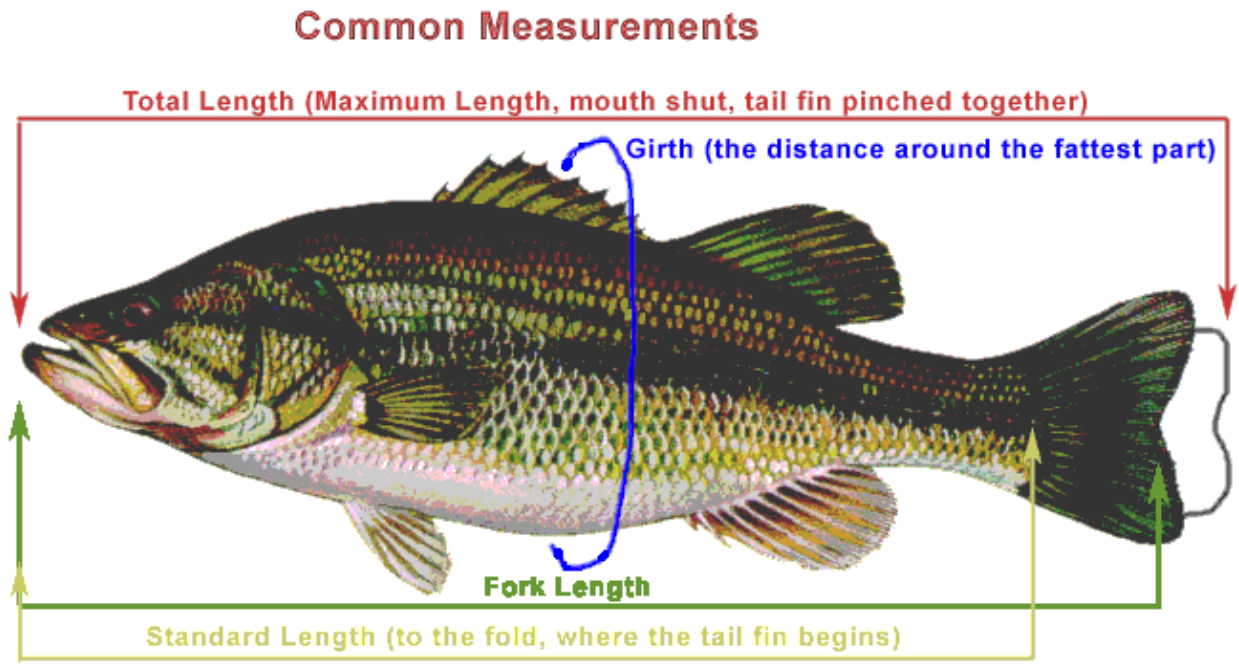
Find the lateral line located laterally at mid-body running from head to tail. It arches dorsally over the operculum.

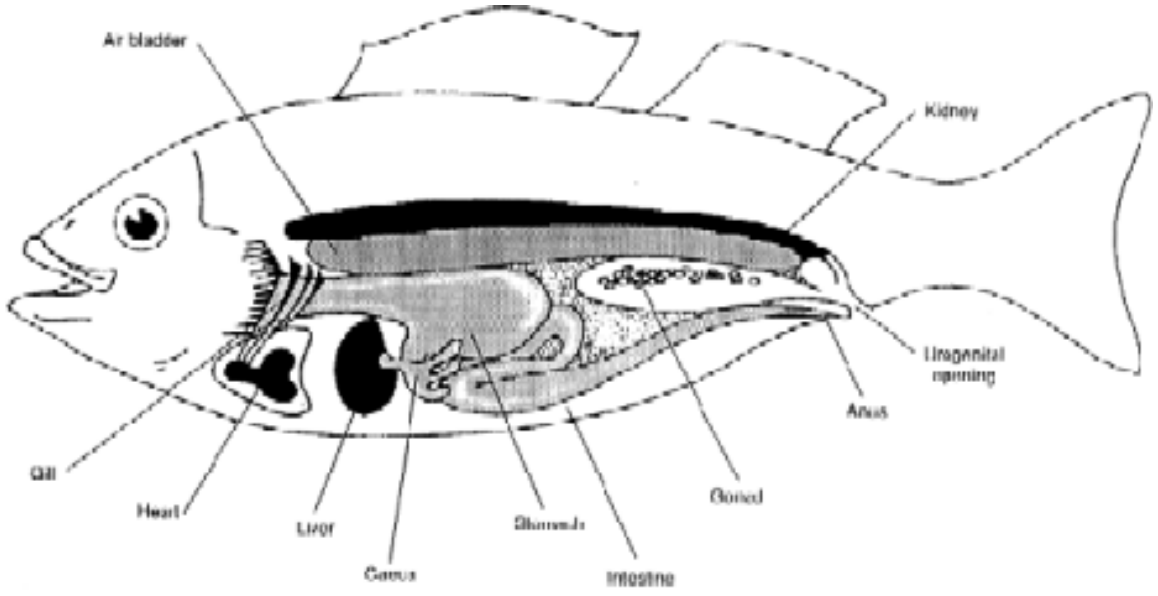


Attachment III: Typical Measurement Locations



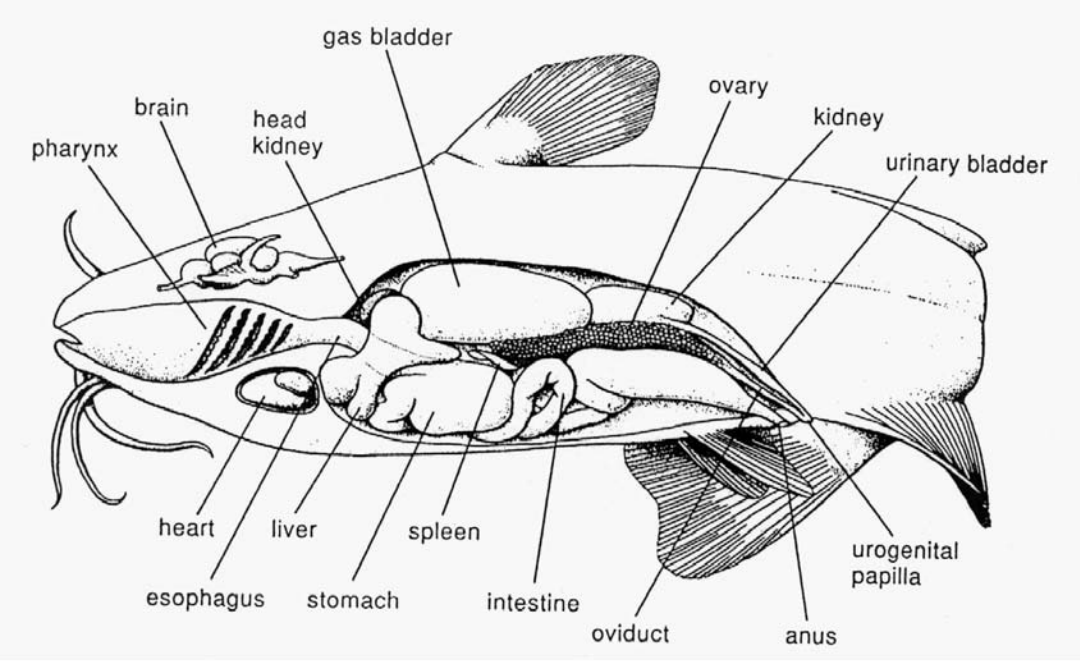
Attachment IV: Typical Measurements of Largemouth Bass





Attachment V: Internal Anatomy

Locate the gonads, either ovaries or testes. Ovaries will appear as numerous spherical structures that may comprise up to 70% of body weight. Testes may comprise up to 12% of body weight. In mature animals they will appear as a soft white organ suspended from the dorsal body wall. Also, if you don't see either of these organs, you might be working with an immature specimen. Note body length and compare to literature on the species/specimen you are working with.

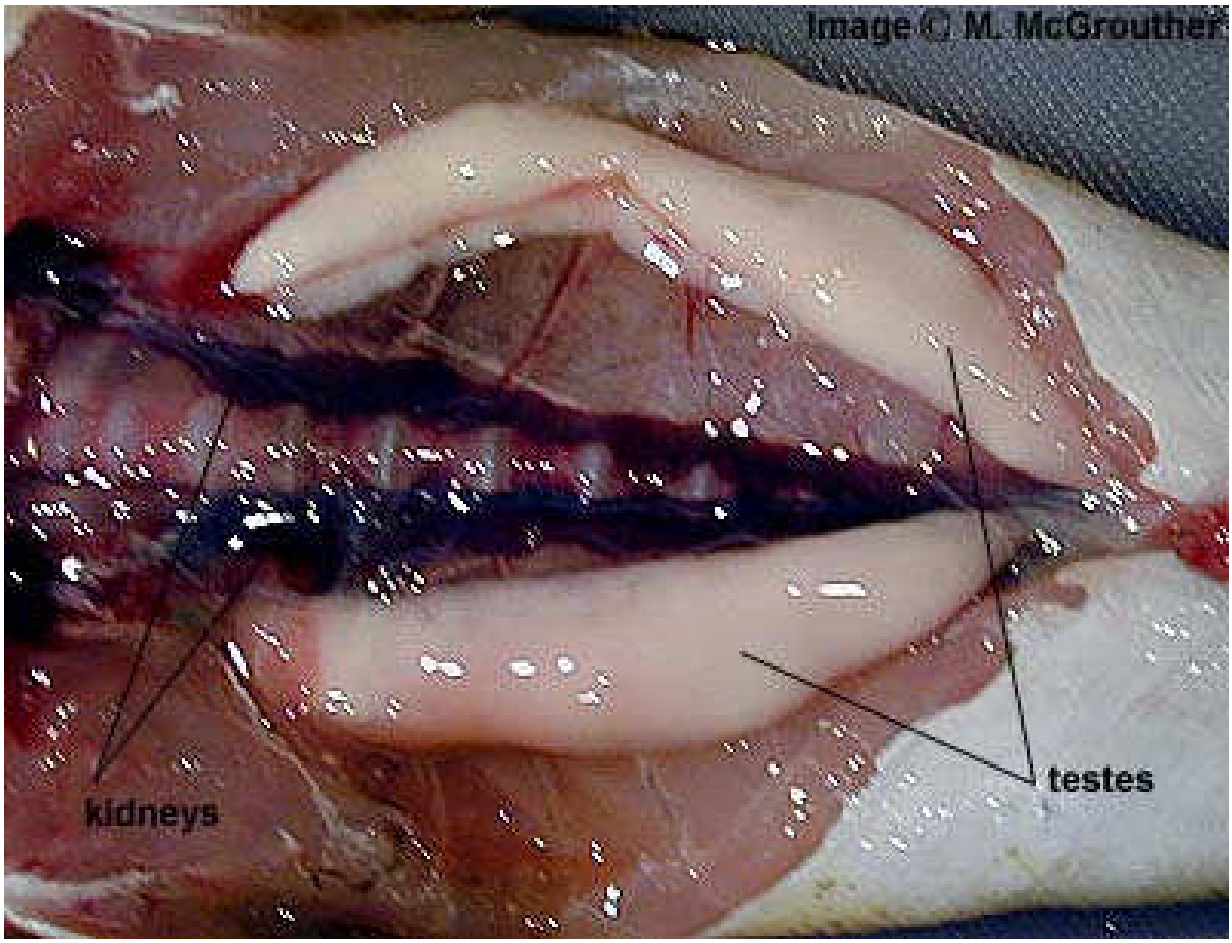


Attachment VI: Kidneys and Testes

Kidneys and Testes: The gonads and kidneys of an Eastern Blue-spotted Flathead. Although Eastern Blue-spotted Flatheads are not collected as part of the Hudson River fish monitoring programs, the image below is representative of the anatomy of fishes typically encountered. The gonads (testes) are the large, pale organs and the kidneys are the red tissue either side of the backbone.

Along the dorsum of the body cavity lies the swim bladder. It is a thick-walled white organ. Occasionally you may see hemorrhages in the swim bladder.

The kidneys also lie in the dorsum of the body cavity. The head kidney and trunk kidney are roughly divided by the swim bladder. In some species (e.g., salmonids) the kidneys are almost fused. The kidneys often exhibit lesions, and the trunk kidney is usually the preferred site for obtaining bacterial and viral cultures. In most fish we work with in this lab, the head kidney and trunk kidney appear fused.



ATTACHMENT 2

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STANDARD OPERATING PROCEDURE

EXTRACTION OF PCBS AND EXTRACTION AND DETERMINATION PERCENT LIPIDS FOR FISH AND BIOTA MATERIALS (NY METHOD)

Reference Methods: EPA Method 3541

SOP Number:	S-GB-O-068-rev.01
Effective Date:	Date of Final Signature
Supersedes:	S-NY-O-017-Rev.14 (Appendix A3-4 of RAM QAPP Attachment A)

APPROVALS

Nils Melberg, General Manager — Date: 01/31/18

Kate E. Verbeten, Quality Manager — Date: 1/30/18

Chris Haase, Department Manager — Date: 1/30/18

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

This is **COPY#** _____ Distributed on _____ by _____ and is ____ **CONTROLLED**

TABLE OF CONTENTS

1. Purpose/Identification of Method

1.1. This is a Standard Operating Procedure (SOP) for the extraction of polychlorinated biphenyls from fish material by SW-846 Method 3541 (Automated Soxtherm Extraction) for subsequent analysis by SW-846 Method 8082A. This SOP also provides the procedures for the extraction and determination of Lipid content (% Lipids). Lipid Content is not a certifiable analyte by NYSDOH.

1.2. The purpose of this SOP is to provide the technician with the procedures required to perform the extraction of PCBs, in biota material samples, using the Automated Soxtherm extraction technique and to perform the subsequent extract volume reduction.

2. Summary of Method

2.1. Samples are initially dried utilizing sodium sulfate.

2.2. The samples are then loaded into the automated Soxtherm extraction beakers where they are spiked and surrogated.

2.3. After a 2 hour extraction the derived solvent is exchanged to pure hexane via a Turbo Vap or PowerVap evaporator system.

2.4. The extract may be put through cleanup processes (see separate SOPs) and is then properly diluted and submitted for GC analysis.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP are applicable to all personnel involved in the Soxtherm extraction of fish.

3.2. The following procedure is used by Pace Analytical Services, LLC. for the extraction of fish tissue for PCB analysis, although it may be adapted for other biota extractions.

3.3. **Parameters**: See analytical methods for analyte lists.

4. Applicable Matrices

4.1. This test method is appropriate for all fish material. The extraction technician must have an understanding of the methods and requirements of USEPA-SW- 846 "Test Methods for Solid Wastes" Volume 1B: Lab Manual, 3rd edition. Methods 3541 and 3500C.

4.2. An approved instructor must also certify the extraction technician for performing the procedure. The extraction technician should have completed an acceptable demonstration of capability before performing this method without supervision. This method may be restricted to use by or under the supervision of an extraction technician knowledgeable in the area of PCB extraction and clean-up. The extraction technician should further be aware of the proper care and handling of PCBs as well. Refer to SDS for details.

5. Limits of Detection and Quantitation

5.1. See determinative method SOPs (S-GB-O-067 latest version) for details relating to the limits of detection and quantitation for PCB analysis.

5.2. A Practical Quantitation Limit for Percent Lipids Analysis (see section 12.4) has been established as 0.03% based upon an analytical balance sensitivity of 0.001 g or better and a nominal sample weight of 10.000 g.

6. Interferences

6.1. Laboratory contamination can occur by the introduction of plasticizers (phthalate esters) into the samples through the use of certain plastics. Phthalate esters respond on electron capture detectors, usually as late eluting peaks, and can interfere in PCB quantification. Samples and extracts should not be exposed to plastics such as gloves, tubing, coating on clamps, and pipette bulbs, etc.

7. Sample Collection, Preservation, Shipment and Storage

7.1. All samples should remain frozen at all times unless being tested. Fish usually arrive whole bodied or already filleted. Once received, the sample must be ground and homogenized so that it may be analyzed. The homogenized fish tissue should be held for 6 to 12 months.

7.2. The fish solvent extracts should be held for 3 months. Some clients may request that the body and/or head of fish be saved once the fillets are cut out.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

8.2. **Homogenize**- to reduce particles so that they are uniformly small, of uniform color and consistency, with no free fluid, and evenly distributed. The portion of sample that has been homogenized is referred to as the homogenate throughout this SOP.

8.3. **Laboratory Control Sample (LCS):** Also known as the Quality Control (QC) Check Standard or Quality Control (QC) Check Sample. The LCS consists of sodium sulfate to which known quantities of the method analytes are added. The LCS is extracted and cleaned up exactly like a field sample, and its purpose is to determine whether the analysis is in control and whether the laboratory is capable of making accurate and precise measurements.

8.4. **Lab Control Sample Duplicate:** An exact copy of the Lab Control Sample to further assess analyte recovery efficiency.

8.5. **Laboratory Method Blank:** A laboratory derived sample consisting of a sodium sulfate that is carried through all extraction and cleanup steps. The laboratory method blank is used to define the level of laboratory analyte background or other interferences that exist in the laboratory environment, the reagents, or extraction apparatus.

8.6. **Matrix Spike (MS):** An aliquot of a field sample that is fortified with known quantities of the method analytes and is carried through all the extraction and cleanup steps. Its purpose is to assess the appropriateness of the method for the matrix by measuring the recovery of the method analytes.

8.7. **Quality Control (QC)**: A set of measures for each sample within an analysis methodology to assure that the process is in control

8.8. **Relative Percent Difference (RPD):**- A measure of precision defined as the difference between two measurements divided by the average concentration of the two measurements.

8.9. **Sample Duplicate**– A second aliquot of a sample that is treated in the same manner as the original sample in order to determine the precision of the method.

8.10. **Sample Matrix Spike Duplicate (MSD):** An exact copy of the Matrix Spike. This is an aliquot of a field sample which is fortified with known quantities of the method analytes and is subject to the entire procedure. Its purpose is to assess the appropriateness of the method for the matrix by measuring the recovery of the method analytes.

8.11. **Surrogate Standard Solution:** The chemical composition and chromatography of surrogates are similar to the analytes of interest. They are usually not found in environmental samples. These compounds are spiked into all samples, blanks, and matrix spike samples prior to extraction. Percent recoveries are calculated for each surrogate.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Soxtherm Extractors with controllers – Gerhardt SE-416

9.2. Analytical Balance: Sargent-Welch 400DR capable of weighing 300±.01grams (or equivalent) used to determine sample mass.

9.3. Chiller: Polyscience Durachill air and water chiller 1.5Hp (or equivalent).

9.4. TurboVap Evaporator: Zymark #ZW640-3.

9.5. PowerVap Evaporator: Fluid Management Systems (or equivalent).

9.6. TurboVap Evaporator concentrator tubes: Zymark 250mL, 0.5mL endpoint.

9.7. Beakers: Assorted Pyrex: 250mL, 600mL, and 1000mL, used for liquid containment and pipette storage.

9.8. Vials: glass, 40mL and 4 dram (with polyseal sealed cap) (20mL and 10mL) capacity, for standards.

9.9. Culture tubes: 15mL and 9mL with Telfon screw caps (T102-3CS and T102-1CS).

9.10. Vial Rack: Plastic rack used to hold vials, during all phases of the extract processing.

9.11. Centrifuge: International Equipment Co., Model CL (or equivalent).

9.12. Pipettes: S/P Disposable Serological Borosilicate Pipettes.

9.12.1. 1mL x 1/10.

9.12.2. 5mL x 1/10.

9.12.3. 10mL x 1/10.

9.13. Disposable Pasteur pipettes MG Scientific 5.75” P200-1 (or equivalent).
9.14. Disposable Pasteur pipettes MG Scientific 9” P200-2 (or equivalent).
9.15. Syringes: 250-1000µL Gastight Syringes (Hamilton 1000 series or equivalent)

9.16. Fiberglass wool: MG Scientific G290-1

9.17. Glass Funnels: HGF

9.18. Aluminum weigh dishes: 60mm weigh pans MG Scientific B-165-2 (or equivalent).

9.19. 4oz. Jars: Industrial Glassware.

9.20. Aluminum foil

10. Reagents and Standards

10.1. Sodium Sulfate: J.T.Baker, #3375-05 Anhydrous, Granular (12-60 mesh) (or equivalent). Used for the laboratory method blank and laboratory control sample.

10.2. Hexane: High Purity Solvent (J.T Baker) #9262-8P (or equivalent).

10.3. Acetone: High Purity Solvent (Burdick/Jackson) #UN1090 (or equivalent).

10.4. 1:1 Hexane/Acetone: 50%/50% by volume solvent mixture prepared in the lab.

10.5. Spike standard solution: PCB Aroclor in Acetone at 20.0µg/mL:

10.5.1. To make a 20.0µg/mL Aroclor spike: Allow the Stock Standard Solution (latest version of SOP S-GB-O-067) to warm up to room temperature. Using a gastight syringe, add 4.0mL of the Stock Standard Solution at 1000µg/mL to a 200mL volumetric flask and set to volume with acetone. All information is recorded in the Standards logbook and LIMS.

10.6. Surrogates Standards: Tetra-chloro-meta-xylene (TCMX) and Decachlorobiphenyl (DCB) TCMX/DCBP at 2.0ug/mL in Acetone:

10.6.1. To make this standard an ampule of is brought to room temperature and shaken on a wrist action shaker for at least 30 minutes. Once the standard is room temperature 1.0mL of a 2000µg/mL solution is added to a 1000mL volumetric flask and set to volume with acetone. All information is recorded in the Standards logbook and LIMS.

11. Calibration and Standardization

11.1. The analytical balance should be calibrated daily to ensure accurate measurements are made when weighing out samples for extraction.

11.2. Please see determinative method (latest version of SOP S-GB-O-067) for details.

12. Procedure

12.1. **Sample Preparation**:

12.1.1. Throughout the entire process it should be noted that if the extraction technician encounters any problems or difficulties with any samples or steps involved, all work should STOP! Any problems should be brought to the attention of the supervisor and documented in LIMS.

12.1.2. The extraction technician should match each sample container label to the chain of custody identification number which is in the job folder.

12.1.3. The fish samples are usually received as fillets and must be processed to produce a homogenous material prior to extraction. Once the sample has been logged into LIMS, the sample is processed according to the latest version of SOP S-GB-L-.

12.1.4. The sample is then placed into the freezer for storage until the extraction process is started.

12.2. **Procedure: Sample Extraction:**

12.2.1. Rinse the Soxtherm extraction beakers with Hexane. Label with sample identification.

12.2.2. Prior to weighing out sample for extraction, the sample must be thoroughly homogenized by mixing the sample in the jar until the skin is equally distributed throughout the tissue and the sample is of uniform color and consistency, and there is no free fluid. Weigh approximately 10g of sample into a Soxtherm beaker with a metal spatula, being careful to maintain an equal skin-tissue ratio in the aliquot to be extracted as the ratio in the original sample jar. At this point, remove spatula used to weigh sample and place it in jar labeled "dirty spatulas". Record the mass of the sample in the electronic prep log to the nearest tenth of a gram. Repeat the process for all samples and quality control samples. **Note:** All sample containers are to be returned to the freezer.

12.2.3. Add enough anhydrous sodium sulfate to sufficiently cover the sample and mix well with a clean metal spatula. If the sample has not dried after a few minutes, more sodium sulfate may be added. Once the sample is well-dried and free flowing, transfer the sample to a pre-labeled extraction beaker using the same metal spatula. **Note:** Be careful not to add too much drying agent to the sample, if too much is added, the sample may not fit completely into the beaker. In this case the sample will have to be split into two separate Soxtherm apparatus set-ups and re-combined following extraction. Spike the beaker with 250µL of 2.0µg/mL surrogate spiking solution. Apply directly to the dried sample.

12.2.4. Spike each laboratory control spike (LCS) and matrix spike (MS/MSD) with 125µL of 20.0µg/mL PCB matrix spike solution.

12.2.5. Add approximately 140mL of a 1:1 hexane/acetone mixture.

12.2.6. Verify the automated Soxtherm extraction settings as summarized.

Extraction temperature	180°C
Boil Time	2 hours
Solvent Reduction	0
Extraction Time	0
Cycle Time	2 hours
Solvent	1:1 Hexane:Acetone

12.2.7. Place the Soxtherm beakers into the Soxtherm unit. Rotate the extraction beakers slightly to ensure a proper seal of the top o-ring and start the extraction procedure. The process will produce approximately 100 mL of extract.

12.2.8. Then, turn on chiller to cool the condensers. Chiller should be set to approximately 12°C.

12.2.9. Prepare filtering funnels by adding a small plug of fiber glass wool into the neck of the funnel. A small wooden stick may need to be used to properly place the glass wool plug. Add approximately 9g of sodium sulfate to funnels. All samples including QC samples must be dried through sodium sulfate.

12.2.10. Pre-rinse TurboVap tubes with Hexane and allow them to dry. Using an individual TurboVap Tube stand, label a TurboVap Tube with the corresponding sample ID number and place in the holder. Place funnel on the TurboVap tube and rinse with Hexane.

12.2.11. After extraction is complete, transfer extract to the TurboVap tube through the funnel to separate any free flowing sample. Once the extract has completely passed through the funnel, take a spatula and transfer the sample from the extraction beaker to the funnel and rinse the beaker with Hexane.

12.2.12. When the funnel goes dry, add enough Hexane to completely saturate the sample in the funnel. Let the solvent drain until the funnel is dry.

12.2.13. All glassware must be rinsed with acetone and dried in the hood before being cleaned as per the latest version of SOP S-GB-O-015.

12.3. **Solvent Reduction: TurboVap/PowerVap:**

12.3.1. The TurboVap or PowerVap evaporator system is used in place of the Kuderna Danish (KD)-concentrator apparatus. The TurboVap evaporator system is used to reduce the sample volume. The TurboVap uses a heated water bath and positive pressure nitrogen flow / vortex action. The PowerVap uses a heated aluminum block as well as positive pressure nitrogen flow/vortex action. Both units maintain a slight equilibrium imbalance between the liquid and gaseous phase of the solvent extract, which allows fractional reduction of the solvents without loss of higher boiling point analytes.

12.3.2. Turn the unit on and allow it to heat up to 40°C ± 2°C. If using the PowerVap, turn on and allow it to heat up to 60 ± 2°C

12.3.3. As a precaution both system regulators should be checked to assure that there is no residual gas pressure within the system and that the gas pressure regulator is off before placing samples in the apparatus. Residual gas pressure may cause splashing and cross contamination of samples. To bleed the system of residual gas pressure place an empty TurboTube into the water bath and close the lid. Make sure that the nitrogen gas pressure regulator is off. Bleed any residual gas until the regulator gauge reads "0" psi. Remove the empty TurboTube.

12.3.4. Rinse each tip and wipedown surfaces of the TurboVap/PowerVap with solvent. Close the lid and turn up the pressure to blow the lines clean. Turn off the pressure and bleed the system of any residual gas.

12.3.5. Place TurboTubes containing the sample extract into the TurboVap/PowerVap and close the lid. Slowly open the pressure regulator. Keep the gas pressure very low, until the solvent level is decreased, to avoid splashing. Increase the gas pressure as the sample reduces, maintaining uniform flow throughout the volume reduction.

12.3.6. The process for solvent (hexane/acetone) reduction takes approximately 20-30 minutes. Do not leave the unit unattended as extracts may be blown to dryness and PCB loss may occur. Immediately notify a supervisor if an extract is blown to dryness. Note if any tubes are blown down to dryness in LIMS.

12.3.7. Concentrate the solvent to approximately 10mL. NOTE: Samples may concentrate at different rates and will need to be removed at different times.

12.3.8. Once a sample reaches 10mL, remove the samples from the TurboVap/PowerVap and place in the TurboTube rack. The remaining solvent will consist largely of hexane since the acetone component is fractionally removed at a faster rate than hexane. However, a solvent exchange with hexane should be completed to ensure the acetone has been entirely removed.

12.3.9. Once all of the samples have reached 10mL, fill each TurboTubeback up to approximately 100mL with hexane. Place the samples back into the TurboVap/PowerVap.. Concentrate the solvent back to 2.5mL. Remove the TurboTube and place in a rack. **NOTE**: Not all samples will evaporate at the same rate; sample extracts containing large amounts of petroleum or other non-volatile liquids may stop reducing before the 5mL point is achieved. Samples, which stop reducing, should be removed as soon as possible.

12.3.10. Quantitatively transfer the sample extract into a 10mL culture tube. Rinse the TurboTube with ~2.5mL Hexane, and then transfer the Hexane rinse to the culture tube. Repeat the ~2.5mL hexane rinse to bring the solvent level up to 5mL using a standard reference vial for comparison. No more hexane should be used in the second rinse than is necessary to bring the final volume to 5mL.

12.3.11. All glassware must be rinsed with acetone and dried in the hood before being cleaned as per the latest version of SOP S-GB-O-015.

12.4. **Percent Lipid Analysis:**

12.4.1. Percent lipid analysis is determined on a separate aliquot of the sample extract and cannot be determined on any extract that has been subjected to extract cleanups

12.4.2. Label one aluminum weigh dish for each sample and the batch method blank. A Sample Duplicate will be analyzed and a relative percent difference (RPD) calculated for each batch of percent lipids analysis if sufficient volume is provided. An extracted matrix spike and matrix spike duplicate can be analyzed for percent lipids in place of a Sample Duplicate to provide a calculated RPD.

12.4.3. Using an analytical balance accurately weigh each aluminum dish to the nearest 0.0001g and record in the electronic prep log.

12.4.4. Using a pipette, remove 1mL of the 5mL sample completed in step 12.3.8, and place in the aluminum dish.

12.4.5. Set the dishes aside in the chemical fume hood for solvent evaporation. These need to be set in an area where they will not be disturbed. Be careful not to work over the aluminum dishes. Doing so may get particles into the dishes and affect the results of the percent total lipids.

12.4.6. Weigh the dish a final time and enter the weight into the electronic prep log.

12.4.7. See Section 14 for instructions on calculating % lipids and the relative percent difference (RPD).

12.5. **Sample Extract Cleanup:**

12.5.1. Most extracts of environmental samples that are to be analyzed for PCBs by gas chromatography with electron capture detection, contain co-extracted interfering substances which must be removed before accurate chromatographic analysis can be performed.

12.5.2. See separate cleanup SOPs for details (S-GB-O-034 and S-GB-O-036, as applicable).

13. Quality Control

13.1. The extraction technician should have completed an acceptable demonstration of capability before performing the method without supervision. The addition of spiking material to a sample or blank must be witnessed by another extraction chemist and signed in LIMS. All surrogates and matrix spikes must meet acceptable QC limits.

13.2. A method blank sample and lab control sample must be prepared per each extraction batch or 1 per 20 samples, whichever is more frequent. A matrix spike/lab duplicate should be prepared for every 20 site samples or as per client specified quality assurance project plan (QAPP). The spike default for LCS, MS is .125mL of A1242 at 20µg/mL in acetone. Client and/or project specifications may dictate alternate amount or Aroclor.

13.3. PCB Surrogates TCMX and DCBP are added to each sample prior to extraction to measure extraction/cleanup efficiency. Default surrogate is: 0.25mL of 2.0ug/mL TCMX / DCB in acetone. Client and/or project specifications may dictate alternate amount.

14. Data Analysis and Calculations

14.1. Fish and other biota samples typically require a percent lipid analysis, see Section 12.4. The lipid content is calculated utilizing the below equation:

$$\% \text{ Lipids} = \frac{(\text{Final Weight} - \text{Initial Weight})}{\text{Sample Weight}} \times \frac{\text{Whole Volume (5mL)}}{\text{Extracted Volume (1mL)}} \times 100$$

14.2. The Relative Percent Difference (RPD) for duplicate results must be ≤20%. Calculate the RPD as follows:

$$\%RPD = (S1-S2)*100\%/((S1+S2)/2)$$

Where: S1 = %Lipid for Sample
S2 = %Lipid for Sample Duplicate

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. See determinative method (the latest version of SOPS-GB-O-067) for details.

15.2. For % lipid the duplicate the RPD should be <20%, or project specified criterion.

16. Corrective Actions for Out-of-Control Data

16.1. See determinative method (the latest version of SOP S-GB-O-067) for details.

16.2. For % lipid the duplicate the RPD should be <20% or project specified criterion. If the RPD is exceeded, check all calculations and review procedures, including sample homogenization, for potential errors. If client specifications require it or if potential errors are found, re-extract and re-analyze samples; otherwise report and flag data accordingly.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. See determinative method SOPs (S-GB-O-067 latest version) for details.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. See determinative method SOPs (S-GB-O-067 latest version) for details.

19. Method Modifications

19.1. Not applicable to this SOP.

20. Instrument/Equipment Maintenance

20.1. Not applicable to this SOP.

21. Troubleshooting

21.1. Not applicable to this SOP.

22. Safety

22.1. The technician should have received in-house safety training and should know the location of first aid equipment and the emergency spill/clean-up equipment, before handling any apparatus or equipment.

22.2. Safety glasses, a lab coat and gloves must be worn when handling glassware and samples.

22.3. Polychlorinated biphenyls have been tentatively classified as known or suspected carcinogens. The chemist must review the Safety Data Sheets (SDS) for PCBs and all reagents used in the procedure before handling them. All equipment and solvents should be handled within a lab fume hood.

23. Waste Management

23.1. See the latest version of SOP S-GB-W-001 for details.

24. Pollution Prevention

24.1. The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, “Quality Systems”- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, “Quality Systems”- most current version.

25.4. U.S. EPA SW-846 "Test Methods for Evaluating Solid Waste; Volume 1B Laboratory Manual Physical/Chemical Methods", Office of Solid Waste and Emergency Response, Third Edition, Final Update III, December 1996.

25.5. "Guide to Environmental Analytical Methods", Third Edition, Genium Publishing Corporation, 1996.

25.6. Pace SOP S-GB-O-067 “Determination of PCB Aroclors (NY Method) - EPA 8082A”.

25.7. Pace SOP S-GB-L-009 “Tissue Preparation?Homogenization of Biota and Plant Matrices for GE Hudson River Monitoring Programs”.

25.8. Pace SOP S-GB--015 “Glassware Cleaning”.

25.9. Pace SOP S-GB-O-039 “Copper Cleanup for the Removal of Sulfur from PCB and Toxaphene Samples -EPA 3660B”.

25.10. Pace SOP S-GB-O-034 “Sulfuric Acid Cleanup- EPA 3665A”.

25.11. Pace SOP S-GB-O-036 “Florisil Cleanup- EPA 3620C”.

25.12. Pace SOP S-GB-W-001 “Waste Handling and Management”.

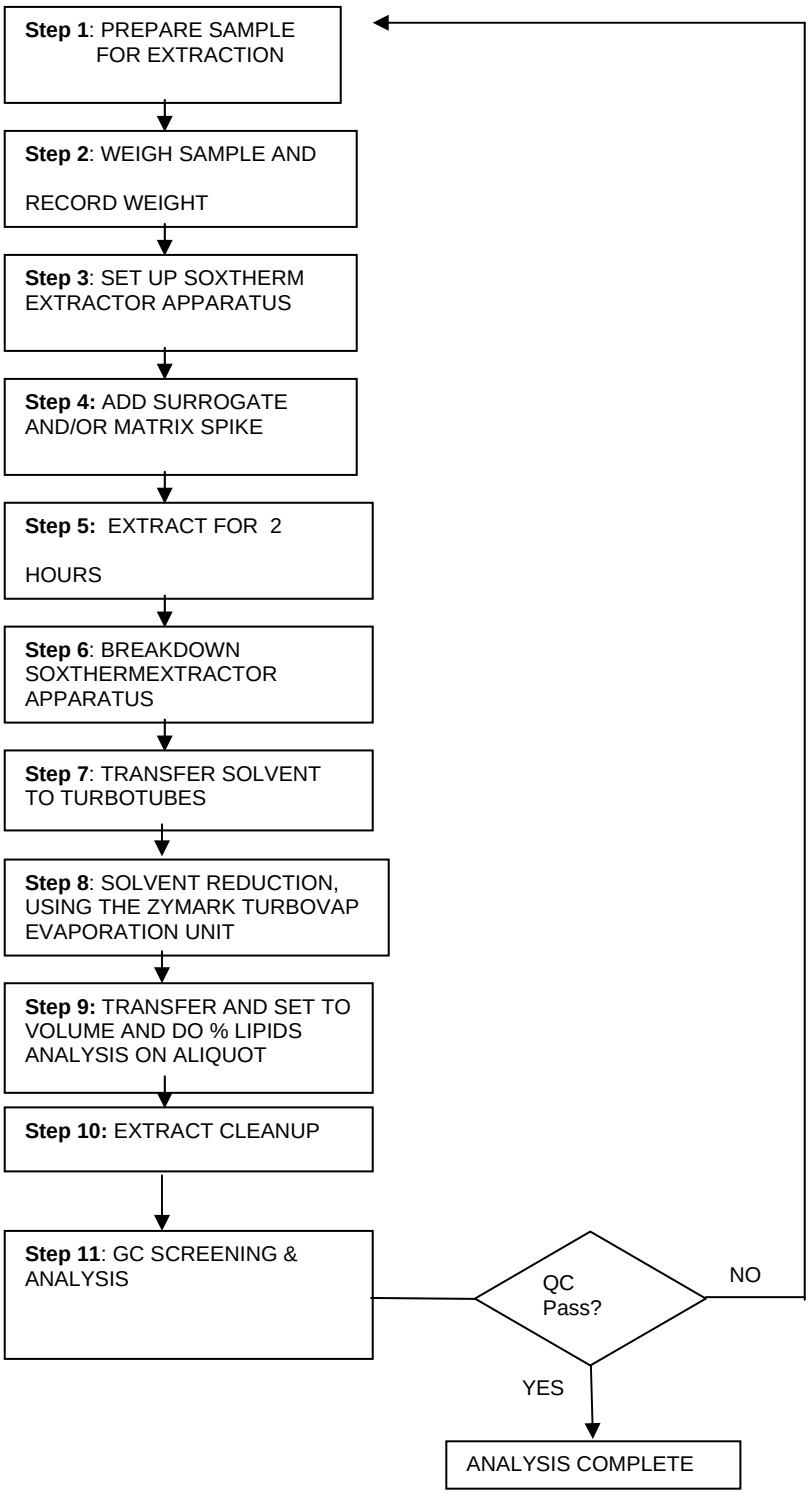
26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I: Flowchart for the Extraction and Clean-up of Fish and Biota Materials for PCB Analysis.

27. Revisions

Document Number	Reason for Change	Date
S-GB-O-068-Rev.00	Used Pace – Schenectady Laboratory SOP S-NY-O-017 to create local SOP.	14Jul2017
S-GB-O-068-Rev.01	Cover page: Updated Supersedes Section to: S-NY-O-017-Rev.14 (Appendix A3-4 of RAM QAPP Attachment A). Section 8: Alphabetized order of defined words in Section. Section 10.5: Changed from 50 to 20µg/mL	29Nov2017

Attachment I: Flowchart for the Extraction and Clean-up of Fish and Biota Materials for PCB Analysis



ATTACHMENT 3

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STANDARD OPERATING PROCEDURE

DETERMINATION OF POLYCHLORINATED BIPHENYL (PCB) AROCLORS

(NY METHOD)

Reference Methods: EPA Method 8082A

SOP NUMBER:	S-GB-O-067-REV.01
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-NY-O-314-Rev.03 (Appendix A3-1 of 2016 RAM QAPP Attachment A)

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 01/31/18

Kate Verbeten, Laboratory Quality Manager — Date: 1/30/18

Chris Haase, Department Manager — Date: 1/30/18

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

This is **COPY#** ____ Distributed on _____ by _____ and is ____ **CONTROLLED**

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1. Purpose/Identification of Method

1.1. This Standard Operating Procedure (SOP) is used to determine Polychlorinated Biphenyl (PCB) Aroclors by capillary gas chromatography (GC) with electron capture detection and total Aroclor quantification using EPA SW-846 Method 8082A

2. Summary of Method

2.1. Samples are extracted with a pesticide analytical grade solvent. The extracts are further processed by concentration and a series of clean-up procedures. The sample extracts are then analyzed by injecting onto a gas chromatographic system equipped with an electron capture detector.

2.2. The purpose of this SOP is to provide a detailed written document for quantification of PCBs as Aroclors according to SW-846 Method 8082A specification.

2.3. This SOP provides detailed instructions for gas chromatographic conditions, calibration, and analysis of PCBs as Aroclors by gas chromatography. Sample extraction and cleanup procedures are described separately in additional laboratory Standard Operating Procedures.

2.4. Extensive knowledge of this SOP and EPA Method 8082A is required. The analysis portion of this method should be performed by a skilled chemist or by an analyst trained in the quantification of trace organics by gas chromatography.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP are applicable to all personnel involved in the analysis of PCBs by Method 8082A.

3.2. **Parameters**: The following PCB Aroclors can be determined by this method:

Compound	CAS Number
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5

4. Applicable Matrices

4.1. This SOP is applicable in the determination and quantification of PCBs as Aroclors as outlined in EPA SW-846 Method 8082A. It is applicable to fish tissue samples.

5. Limits of Detection and Quantitation

5.1. The following are default reporting limits as of the effective date of this SOP. Current reporting limits are on file with the QA Department and can be obtained by request.

Matrix	Sample Mass/Volume Extracted	Calibration Curve Low Standard	Extract Volume	RL (PQL) (all Aroclors)
Fish	10g (wet weight basis)	0.10µg/mL	5mL	50µg/kg

6. Interferences

6.1. Laboratory contamination can occur by the introduction of plasticizers (phthalate esters) into the samples through the use of flexible tubing. Samples and extracts should not be exposed to plastic materials. Phthalate esters exhibit response on electron capture detectors, usually as late eluting peaks, and can interfere in PCB quantification. Laboratory method blanks must be thoroughly reviewed for presence of non-target peaks and comparison of samples with blank chromatographic patterns.

6.2. Polychloroterphenyls (PCTs), polybrominatedbiphenyls (PBB), polychlorinated naphthalenes (PCN), as well as dioxins can co-elute with PCBs. Carry-over from these compounds, when in high concentration, is common if clean-up procedures are not followed. These materials may be removed through the use of specified clean-up procedures.

6.3. Pesticides can be a source of contamination through breakdown into components such as hexachlorobenzene (HCB). This chlorinated compound can carry-over on the GC column, and contaminate samples. Specified clean-up procedures should be followed to eliminate this as a source of contamination when analyzing PCBs. High concentrations of pesticides can cause carry-over on GC columns.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Sample Collection and Preservation:

7.1.1. All samples must be placed on ice, refrigerated at ≤6°C, or frozen from the time they are collected until delivery to the lab.

7.2. Sample Shipment:

7.2.1. Sample Shipment is accomplished through a carrier such as Federal Express or United Postal Service for overnight 1-day delivery to the lab. Shipment is normally handled by the field personnel collecting the samples and coordinated with sample receiving department at the lab. Samples can also be picked up by the lab courier service if samples are collected within driving distance to the lab.

7.3. Sample Storage:

7.3.1. The samples must be protected from light and frozen at ≤-10°C from time of receipt until they are removed from storage for extraction. Remaining sample material will be stored protected from light and frozen at ≤-10°C. Sample will be disposed of or stored / archived according to project specifications.

7.4. Sample Extract Storage:

7.4.1. Sample extracts must be protected from light and refrigerated at ≤6°C. Sample extracts can be discarded after 45 days from issuance of final deliverables or can be archived in a freezer at ≤-10°C for longer periods of time depending on the program requirements.

7.4.2. Field samples, sample extracts, and calibration standards must be stored separately.

7.5. Required Hold Time:

7.5.1. Extraction of tissue samples by appropriate technique must be completed within one year from sample collection. Sample extracts must be analyzed within forty days from extraction.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

8.2. **Accuracy** – The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations; a data quality indicator.

8.3. **Analytical Batch** – An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed 20 samples.

8.4. **Blank** – A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.

8.5. Initial Calibration Verification Standard (ICVs) –The initial calibration verification standard contains all target analytes found in the calibration standards and is used to verify that the initial calibration is prepared correctly and that the instrument system is correctly calibrated. Initial Calibration Verification Standard solutions are made from a stock solution which is different from the stock used to prepare the initial calibration standards.

8.6. **Continuing Calibration Verification Standard (CCVs)** –The continuing calibration verification standard contains all target analytes found in the calibration standards and is used to verify that that the instrument system is correctly calibrated and good working order. Calibration check solutions are made from the same stock solution used to prepare the initial calibration standards.

8.7. **Calibration Standard** – A substance or reference material used for calibration.

8.8. **CAS Number** – An assigned number used to identify a chemical. CAS stands for Chemical Abstracts Service, an organization that indexes information published in Chemical Abstracts by the American Chemical Society and that provides index guides by which information about particular substances may be located in the abstracts. Sequentially assigned CAS numbers identify specific chemicals, except when followed by an asterisk (*) which signifies a compound (often naturally occurring) of variable composition. The numbers have no chemical significance. The CAS number is a concise, unique means of material identification. (Chemical Abstracts Service, Division of American Chemical Society, Box 3012, Columbus, OH 43210: [614] 447-3600).

8.9. **Duplicate**– The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results of duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory.

8.10. **Environmental Sample** – An environmental sample or field sample is a representative sample of any material (aqueous, non-aqueous, or multimedia) collected from any source for which determination of composition or contamination as requested or required.

8.11. **Initial Calibration** – The process of analyzing standards, prepared at specified concentrations, to define the quantitative response relationship of the instrument to the analytes of interest. Initial calibration is

performed whenever the results of a calibration verification standard do not conform to the requirements of the method in use or at a frequency specified in the method.

8.12. **Laboratory Control Sample (LCS)** – A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes and taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to evaluate the performance of all or a portion of the measurement system.

8.13. **Laboratory Control Sample Duplicate (LCSD)** – A replicate laboratory control sample prepared and analyzed to obtain a measure of the precision of the recovery for each analyte.

8.14. **Laboratory Method Blank** – A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses..

8.15. **Matrix** – The predominant material of which the sample to be analyzed is composed; the substrate of a test sample. Matrix is not synonymous with phase (liquid or solid).

8.16. **Matrix Spike** – A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency..

8.17. **Matrix Spike Duplicate** – A replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

8.18. **Method Detection Limit (MDL)** – The minimum measured concentration of a substance that can be reported with 99% confidence that the measured concentration is distinguishable from method blank results.

8.19. **SDS – Safety Data Sheet**. OSHA has established guidelines for the descriptive data that should be concisely provided on a data sheet to serve as the basis for written hazard communication programs.

8.20. **PCB- Polychlorinated biphenyls** are a class of 209 individual chemical compounds (congeners), in which one to ten chlorine atoms are attached to biphenyl. Use of PCBs has made them a frequent environmental pollutant.

8.21. **Precision** – The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. Precision is assessed by means of duplicate/replicate sample analysis.

8.22. **Practical Quantitation Limit (PQL) -** Another term for a method reporting limit. The lowest reportable concentration of a compound based on parameters set up in an analytical method and the laboratory's ability to reproduce those conditions

8.23. **Quality Control** – The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality.

8.24. **Stock Solution** – A concentrated reference solution containing one or more analytes prepared in the laboratory using an assayed reference compound or purchased from a reputable commercial source.

8.25. **Surrogate** – A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Gas Chromatograph: Complete system for high resolution, capillary column capability and all required accessories. Pace Analytical Services, Inc. will use an Agilent 6890 (or equivalent) gas chromatograph (or equivalent), equipped with an Agilent 7683B autosampler injector (or equivalent), and micro-electron capture detector (or equivalent).

9.2. Chromatographic Data System: A data system for measuring peak height and peak area. Chemstation software will be employed to capture detector response and digitally store the chromatographic data files. Target processing software will be utilized for electronic peak integration for precise calculations and database structuring of the analytical information.

9.3. Column (Primary Hydrogen Carrier Gas): ZB-1MS, Phenomenex Cat. No 7FD-G011-08; 20m x 0.18mm x 0.18um.

9.4. Hamilton Gastight syringes: 0.010 – 1.0mL

9.5. Class A pipettes: 1.00 – 100.0mL

9.6. Class A volumetric flasks10, 50, 100 and 200mL.

9.7. 4 mL glass vials for sample extract storage.

9.8. 125mL amber vial Thermo Scientific 349-0125 and 20mL amber vial Thermo Scientific B7921-VO.

9.9. Pasteur pipettes.

9.10. 250mL and 100mL beakers, glass.

9.11. Hexane, J.T. Baker-Pest Grade (or equivalent).

9.12. Acetone, Burdick and Jackson.-Pest Grade (or equivalent).

9.13. Ferrules: 0.4mm graphite/vespel, Restek 20229, and ¼" graphite ferrules, Restek 20210 or equivalent.

9.14. Injector septa: Teflon Faced septa, Supelco 22731 or equivalent.

9.15. Injector liner: Gooseneck Splitless Liner, Restek 20799 or equivalent.

9.16. Hamilton injector syringe 10.0µL: 1481528 or equivalent.

9.17. Auto sampler vials: 12x32mm Clear National Scientific C4011-1 (or equivalent).

9.18. Snap Caps: 11mm Aluminum Seal SF type PTFE, National Scientific C4011-7A (or equivalent).

10. Reagents and Standards

10.1. Aroclor Stock Standard Solutions:

10.1.1. Polychlorinated Biphenyls - Stock standards are prepared from individual Aroclor stock solutions. See Table 10.1 below for the exact preparation of each compound. Purchased stock standards should be stored according to manufacturer recommendations.

10.1.2. The stock standards are transferred into screw-cap Boston bottles and stored under refrigeration at ≤6°C protected from light. Stock standards should be checked frequently for signs of evaporation, especially just prior to preparing calibration standards. Stock PCB standards must be replaced after one year or sooner if a problem with instrument calibration is detected.

Table 10.1: Aroclor Stock Standards

Standard	Concentration	Manufacturer*	Catalog #	Storage	Expiration Date
Pesticide Surrogate Mix	200µg/mL each in Acetone	Restek Corporation	32000	Refrigerate ≤6°C	Manufacturer's recommended expiration date for unopened ampulated standards. Stock standards must be replaced 1 year after ampule is opened or on expiration date, whichever is sooner
Aroclor 1221 Mix	1000µg/mL in Hexane	Restek Corporation	32007	Ambient	
Aroclor 1232 Mix	1000µg/mL in Hexane	Restek Corporation	32008	Ambient	
Aroclor 1242 Mix	1000µg/mL in Hexane	Restek Corporation	32009	Ambient	
Aroclor 1248 Mix	1000µg/mL in Hexane	Restek Corporation	32010	Ambient	
Aroclor 1254 Mix	1000µg/mL in Hexane	Restek Corporation	32011	Ambient	
Aroclor 1016/1260 Mix	1000µg/mL in Hexane	Restek Corporation	32039	Ambient	
	1000µg/mL in Hexane	O2sI		Ambient	
Aroclor 1016	1000µg/mL in Isooctane	Supelco	4-8097	Ambient	
Aroclor 1260	1000µg/mL in Isooctane	Supelco	4-4809	Ambient	
Aroclor 1268	1000µg/mL in Hexane	Restek Corporation	32410	Ambient	

*Or Equivalent

10.2. Intermediate Stock Standards: The following Table 10.2 is a listing of the prepared intermediate stock standards.

Table 10.2: Intermediate Stock Standards

Analytical Standard	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Expiration Date
TCMX/DCB Stock Solution	Pesticide Surrogate Mix	1000µL	20mL of Hexane	10µg/mL	1 year from date of preparation; or Manufacturer's expiration date, whichever is sooner.
AR1221 Stock Solution	Aroclor 1221 Mix	1000µL	10mL of Hexane	100µg/mL	
AR1232 Stock Solution	Aroclor 1232 Mix	1000µL	10mL of Hexane	100µg/mL	
AR1242 Stock Solution	Aroclor 1242 Mix	1000µL	10mL of Hexane	100µg/mL	
AR1248 Stock Solution	Aroclor 1248 Mix	1000µL	10mL of Hexane	100µg/mL	
AR1254 Stock Solution	Aroclor 1254 Mix	1000µL	10mL of Hexane	100µg/mL	
AR1660 Stock Solution	Aroclor 1016/1260 Mix	1000µL	10mL of Hexane	100µg/mL each	
AR 1268 Stock Solution	Aroclor 1268 Mix	1000µL	10mL of Hexane	100µg/mL each	
AR1660 ICV Stock Solution	Aroclor 1016 Aroclor 1260	1000µL each	10mL of Hexane	100µg/mL each	

10.3. Calibration Standards:

10.3.1. Calibration standards are prepared at five concentration levels using a prepared intermediate stock standard. See Table 10.3 and for the preparation and exact concentrations of the working standards. The following five standards make up the initial calibration: 0.10µg/mL, 0.2µg/mL, 0.50µg/mL, 0.8µg/mL, 1.0µg/mL.

10.3.2. The two surrogates Tetra-chloro-meta-xylene (TCMX) and Decachlorobiphenyl (DCB) are included in all calibration standards..

10.3.3. Refer Table 10.3 for instructions on preparation of the calibration standards containing the surrogates.

10.3.4. Transfer all calibration standards to 125mL amber vials and store in a refrigerator at ≤6°C, protected from light. Calibration standards must be replaced after six months, or sooner, if comparison with check standards indicates a problem

10.3.5. Calibration Verification:

10.3.6. Initial Calibration Verification (ICV) – In order to consider the initial calibration acceptable, an ICV standard must be analyzed for each 5 point calibration. The ICV standard must be from a second source stock and meet the same criteria as the continuing calibration verification standard before the initial calibration may be considered valid.

10.3.7. Continuing Calibration Verification (CCV) – A midpoint calibration check standard must be injected at the beginning and end of each 12-hour analysis period, and at intervals of not less than once every 10 samples, for calibration verification. If the response factor (area/concentration) of the check standard deviates by more than 20% from the initial average response factor, the calibration is considered out of control and analysis must be stopped.

10.3.8. Acceptance Criteria – The percent difference (%D) is determined for every analyte and must be within ±20% of the calibration curve. Calculate %D for each peak using the following equation:

$$\%D = \left(\frac{R_1 - R_2}{R_1} \right) x100$$

Where:

R1 = Mean Response factor from the ICAL

R2 = RF calculated from the CCV

10.3.9. The %D for all of the peaks used for quantitation must be < 20% to meet acceptance criteria. If the Aroclors themselves are acceptable, evaluate the %D for each surrogate. If the %D is ≤ 20% for each individual Aroclor and surrogate, the continuing meets the acceptance criteria.

Table 10.3: Analytical Standards

Analytical Standard	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Expiration Date
AR1221-1 Calibration Standard and PRLS	AR1221 Stock Solution TCMX/DCB Stock Solution	AR1221 100µL TCMX/DCB 100µL	100mL of Hexane	AR1221 0.10µg/mL TCMX/DCB 0.01µg/mL	6 mo. from date of preparation or the expiration date listed for the stock source, whichever is sooner
AR1221-2 Calibration Standard	AR1221 Stock Solution TCMX/DCB Stock Solution	AR1221 200µL TCMX/DCB 200µL	100mL of Hexane	AR1221 0.2µg/mL TCMX/DCB 0.02µg/mL	
AR1221-3 Calibration Standard	AR1221 Stock Solution TCMX/DCB Stock Solution	AR1221 1000µL TCMX/DCB 1000µL	200mL of Hexane	AR1221 0.5µg/mL TCMX/DCB 0.05µg/mL	
AR1221-4 Calibration Standard	AR1221 Stock Solution TCMX/DCB Stock Solution	AR1221 800µL TCMX/DCB 1000µL	100mL of Hexane	AR1221 0.8µg/mL TCMX/DCB 0.10µg/mL	
AR1221-5 Calibration Standard	AR1221 Stock Solution TCMX/DCB Stock Solution	AR1221 1000µL TCMX/DCB 1500µL	100mL of Hexane	AR1221 1.0µg/mL TCMX/DCB 0.15µg/mL	
AR1232-3 Calibration Standard	AR1232 Stock Solution TCMX/DCB Stock Solution	AR1232 500µL TCMX/DCB 500µL	100mL of Hexane	AR1232 0.5µg/mL TCMX/DCB 0.05µg/mL	

Analytical Standard	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Expiration Date
AR1242-1 Calibration Standard and PRLS	AR1242 Stock Solution TCMX/DCB Stock Solution	AR1242 100µL TCMX/DCB 100µL	100mL of Hexane	AR1242 0.10µg/mL TCMX/DCB 0.01µg/mL	6 mo. From date of preparation or the expiration date listed for the stock source, whichever is sooner
AR1242-2 Calibration Standard	AR1242 Stock Solution TCMX/DCB Stock Solution	AR1242 200µL TCMX/DCB 200µL	100mL of Hexane	AR1242 0.2µg/mL TCMX/DCB 0.02µg/mL	
AR1242-3 Calibration Standard	AR1242 Stock Solution TCMX/DCB Stock Solution	AR1242 1000µL TCMX/DCB 1000µL	200mL of Hexane	AR1242 0.5µg/mL TCMX/DCB 0.05µg/mL	
AR1242-4 Calibration Standard	AR1242 Stock Solution TCMX/DCB Stock Solution	AR1242 800µL TCMX/DCB 1000µL	100mL of Hexane	AR1242 0.8µg/mL TCMX/DCB 0.10µg/mL	
AR1242-5 Calibration Standard	AR1242 Stock Solution TCMX/DCB Stock Solution	AR1242 1000µL TCMX/DCB 1500µL	100mL of Hexane	AR1242 1.0µg/mL TCMX/DCB 0.15µg/mL	
AR1248-1 Calibration Standard and PRLS	AR1248 Stock Solution TCMX/DCB Stock Solution	AR1248 100µL TCMX/DCB 100µL	100mL of Hexane	AR1248 0.10µg/mL TCMX/DCB 0.01µg/mL	
AR1248-2 Calibration Standard	AR1248Stock Solution TCMX/DCB Stock Solution	AR1248 200µL TCMX/DCB 200µL	100mL of Hexane	AR1248 0.2µg/mL TCMX/DCB 0.02µg/mL	
AR1248-3 Calibration Standard	AR1248 Stock Solution TCMX/DCB Stock Solution	AR1248 500µL TCMX/DCB 500µL	100mL of Hexane	AR1248 0.5µg/mL TCMX/DCB 0.05µg/mL	
AR1248-4 Calibration Standard	AR1248 Stock Solution TCMX/DCB Stock Solution	AR1248 800µL TCMX/DCB 1000µL	100mL of Hexane	AR1248 0.8µg/mL TCMX/DCB 0.10µg/mL	
AR1248-5 Calibration Standard	AR1248 Stock Solution TCMX/DCB Stock Solution	AR1248 1000µL TCMX/DCB 1500µL	100mL of Hexane	AR1248 1.0µg/mL TCMX/DCB 0.15µg/mL	
AR1254-1 Calibration Standard and PRLS	AR1254 Stock Solution TCMX/DCB Stock Solution	AR1254 100µL TCMX/DCB 100µL	100mL of Hexane	AR1254 0.10µg/mL TCMX/DCB 0.01µg/mL	

Analytical Standard	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Expiration Date
AR1254-2 Calibration Standard	AR1254 Stock Solution TCMX/DCB Stock Solution	AR1254 200µL TCMX/DCB 200µL	100mL of Hexane	AR1254 0.2µg/mL TCMX/DCB 0.02µg/mL	6 mo. From date of preparation or the expiration date listed for the stock source, whichever is sooner
AR1254-3 Calibration Standard	AR1254 Stock Solution TCMX/DCB Stock Solution	AR1254 1000µL TCMX/DCB 1000µL	200mL of Hexane	AR1254 0.5µg/mL TCMX/DCB 0.05µg/mL	
AR1254-4 Calibration Standard	AR1254 Stock Solution TCMX/DCB Stock Solution	AR1254 800µL TCMX/DCB 1000µL	100mL of Hexane	AR1254 0.8µg/mL TCMX/DCB 0.10µg/mL	
AR1254-5 Calibration Standard	AR1254Stock Solution TCMX/DCB Stock Solution	AR1254 1000µL TCMX/DCB 1500µL	100mL of Hexane	AR1254 1.0µg/mL TCMX/DCB 0.15µg/mL	
AR1660-1 Calibration Standard and PRLS	AR1660 Stock Solution TCMX/DCB Stock Solution	AR1660 100µL TCMX/DCB 100µL	100mL of Hexane	AR1660 0.10µg/mL TCMX/DCB 0.01µg/mL	
AR1660-2 Calibration Standard	AR1660 Stock Solution TCMX/DCB Stock Solution	AR1660 200µL TCMX/DCB 200µL	100mL of Hexane	AR1660 0.2µg/mL TCMX/DCB 0.02µg/mL	
AR1660-3 Calibration Standard	AR1660 Stock Solution TCMX/DCB Stock Solution	AR1660 1000µL TCMX/DCB 1000µL	200mL of Hexane	AR1660 0.5µg/mL TCMX/DCB 0.05µg/mL	
AR1660-4 Calibration Standard	AR1660 Stock Solution TCMX/DCB Stock Solution	AR1660 800µL TCMX/DCB 1000µL	100mL of Hexane	AR1660 0.8µg/mL TCMX/DCB 0.10µg/mL	
AR1660-5 Calibration Standard	AR1660 Stock Solution TCMX/DCB Stock Solution	AR1660 1000µL TCMX/DCB 1500µL	100mL of Hexane	AR1660 1.0µg/mL TCMX/DCB 0.15µg/mL	
AR1221-3 ICV Calibration Standard	AR1221 ICV Stock Solution TCMX/DCB Stock Solution	AR1221 500µL TCMX/DCB 500µL	100mL of Hexane	AR1221 0.5µg/mL TCMX/DCB 0.05µg/mL	
AR1242-3 ICV Calibration Standard	AR1242 ICV Stock Solution TCMX/DCB Stock Solution	AR1242 500µL TCMX/DCB 500µL	100mL of Hexane	AR1242 0.5µg/mL TCMX/DCB 0.05µg/mL	

Analytical Standard	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Expiration Date
AR1248-3 ICV Calibration Standard	AR1248 ICV Stock Solution TCMX/DCB Stock Solution	AR1248 500µL TCMX/DCB 500µL	100mL of Hexane	AR1248 0.5µg/mL TCMX/DCB 0.05µg/mL	6 mo. From date of preparation or the expiration date listed for the stock source, whichever is sooner
AR1254-3 ICV Calibration Standard	AR1254 ICV Stock Solution TCMX/DCB Stock Solution	AR1254 500µL TCMX/DCB 500µL	100mL of Hexane	AR1254 0.5µg/mL TCMX/DCB 0.05µg/mL	
AR1660-3 ICV Calibration Standard	AR1660 ICV Stock Solution TCMX/DCB Stock Solution	AR1660 500µL TCMX/DCB 500µL	100mL of Hexane	AR1660 0.5µg/mL TCMX/DCB 0.05µg/mL	

11. Calibration and Standardization

11.1. Gas chromatographic operation parameters: See Attachment III.

11.2. Retention Times:

11.2.1. Retention Time (RT) – Retention time windows are used for compound identifications in samples. The RT for all components in all standards must be within the windows specified for both columns.

11.2.2. Make at least three injections of all analytes of interest over a 72-hour period.

11.2.3. Record the retention time for each selected peak for each Aroclor mixture, to three decimal places. Calculate the mean and standard deviation for each peak.

11.2.4. The width of the retention time window is defined as ± 3 standard deviations of the mean established. The minimum retention window will be + 0.03 minutes.

11.2.5. Establish the center of the RT window for each Aroclor mixture and surrogate using the absolute RT from the calibration verification standard at the beginning of the analytical shift. Optionally, the Initial Calibration RT windows may continue to be used as long as method criteria are met. For samples run during the same shift as an initial calibration, use the RT of the mid-point standard in the Initial calibration as the center of the RT window.

11.3. Retention time studies are performed for each instrument annually and are available upon request form the QA department

11.4. Initial GC Calibration:

11.4.1. GC calibration is performed by the external standard calibration procedure. Prior to running samples the system must be calibrated and system performance must be verified.

11.4.2. Establish the gas chromatographic operating parameters outlined in the Procedure section and prepare the calibration standards at the five concentrations outlined in the Reagent and Standard section. Inject each calibration standard using the GC Autosampler and the parameters outlined in the Procedure section. Note: The same parameters are used for actual samples.

11.4.3. For each Aroclor, 5 peaks are selected to prepare calibration curves. The peaks selected from the multi-component Aroclor formulations were based on maximizing the separation for each Aroclor (i.e., minimizing peak overlap in retention time). Consideration was also given to selecting peaks that normally did not have problems with co-elution with interfering peaks or possible co-elution with organochlorine pesticides. The determined area of the five peaks selected for calibration is processed by the Target workstation for calculations of the calibration factors of each peak. The following table lists the Aroclors that are included in the initial calibration and the peak numbers used.

Aroclor	**Peak Numbers**
A1016	6, 7, 8, 9, 10
A1221	1, 2, 3, 4, 5
A1232	5, 7, 8, 9, 10
A1242	6, 7, 8, 9, 10
A1248	11, 12, 13, 14, 15
A1254	16, 17, 18, 19, 20
A1260	20, 21, 22, 23, 24

11.4.4. The percent relative standard deviation (%RSD) of the five calibration factors for each peak of each Aroclor, along with the surrogates must be ≤20%. If this is the case, linearity can be assumed, and the average RF can be used for quantitation. For linear calibration curve the Correlation Coefficient R must be greater than 0.995.

11.4.5. Response Factors (RF) – Individually tabulate the area responses for each of the five or more peaks selected for each Aroclor versus concentration of the five-point calibration standards for each GC column. Calculate RF for each peak using the following equation:

$$RF = \frac{A_x}{C_x}$$

Where:
A_x = Total area of analyte response.
C_x= Concentration of the analyte in the solution (µg/mL).

11.4.6. Once linear calibration has been established it is subjected to an additional check. This check is the comparison of the calculated amount of the low calibration standard for each 5 point Aroclor against the expected amount of the standard using the % difference. Re-fitting the calibration data back to the model or calculating the % difference is determined by using the following equation:

$$\%Difference = \left(\frac{Cc - Ce}{Ce}\right) X 100\,\%$$

Where: Cc = Calculated amount of standard, in mass or concentration units.
Ce = Expected amount of standard, in mass or concentration units.

The absolute value of the percent difference between these two amounts for every calibration level should be less than or equal to 20%.

11.4.7. If a re-calibration is performed, the ICV must be analyzed again and values calculated using the new relative response factors. If the ICV fails to meet the percent difference criteria after re-calibration, sample analysis must not proceed until the problem is found and corrected (*i.e.,* GC gas leak, autosampler syringe plugged, broken injector liner).

12. Procedure

12.1. Sample Extraction and Preparation may be performed by EPA Methods 3541. See separate extraction SOP for details.

12.2. Gas Chromatographic Procedures:

12.2.1. Prescreening of sample extracts: Prescreening is a fast and effective way to determine if re-extracts or dilutions for over ranged samples are required. See SOP S-GB-O-062 Sample Screening Semi-Volatile Organics Prior to Analysis, current revision for details.

12.2.2. Approximately 1.0mL of the final dilution extract is then transferred into a labeled autosampler vial.

12.2.3. The sequence of the analytical queue is set up in the Chemstation software. This file contains the exact order in which standards, instrument blanks, and samples will be analyzed.

12.2.3.1. Analytical Sequence: The following is an example of the order that initial calibration standards, continuing calibration verification standards, method blanks, QC samples, and samples are placed in an analytical sequence. A continuing calibration verification standard is run after every 10 samples in the analytical sequence. All analytical sequences must end with a continuing calibration verification standard regardless of the number of samples. Below is an example of an analytical sequence.

Order of Material Injected
Hexane Blank
Initial Calibration Standards
Hexane Blank
Initial Calibration Verification
Samples, including method blanks, matrix spikes, matrix duplicates, matrix spike duplicates, and QC reference check standard. A maximum of ten samples between continuing calibration check standards
Continuing Calibration Verification

12.2.4. Once the sample sequence is uploaded into the Chemstation and saved, the sample sequence is printed and the samples are loaded into the GC autosampler tray in the order specified by the sample sequence.

12.2.5. At this point the chromatography software can be initiated to start data collection. The gas chromatograph is placed into run mode and sample analysis is performed until the analytical sequence is complete.

12.2.6. Peak Identification:

12.2.6.1. Target peaks are identified in unknown samples based upon Retention Time (RT). The retention time of an unknown peak must fall within the retention time windows established.

12.2.6.2. Besides using retention time windows to assign peak IDs, the analyst should also rely on their own experience in recognition of multi-response PCB chromatograms. Caution should be exercised when identifying peaks which elute near interferences present in samples and blanks. Comparison of sample chromatograms with method blank and field blank chromatograms is useful in determining chromatographic interferences.

12.2.7 PCB Aroclor Qualitative Identification:

12.2.7.1 Positive identification of PCB Aroclors is based on comparison of retention time of the five selected quantitation peaks and major non-quantitation peaks for the unknown sample with retention time of reference standards (continuing calibration verification). Additionally pattern recognition is used for comparison of unknown samples with reference standards for positive identification.

12.2.7.2 In cases where multiple Aroclors are present with overlapping chromatographic patterns or interferences are encountered that are not removed with extract cleanup processes one or two quantitation peaks may be dropped and not used for quantitation. A minimum of 3 quantitation peaks must be used for all unknown samples and standards

12.3. Data Reduction/Reporting:

12.3.1. Final peak assignments and quantitation calculations are performed within the Target processing software along with the current instrument calibration. The final concentration results are provided in the reporting section of the software.

13. Quality Control

13.1. The table below outlines the data assessment, acceptance criteria, and corrective action procedures for non-compliant data. These are lab default criteria; client specific program requirements may differ from these criteria. Check with the Project Manager, Department Supervisor, or Quality Manager if you are uncertain whether samples being analyzed have client specific requirements.

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration	The five point calibration is analyzed initially for all reported Aroclors and when Continuing Calibration Verification standards fail criteria.	- %RSD≤20% for the relative response factors for the calibration standards if using average response factor calibration. Correlation Coefficient R must be >0.995 for Linear Regression.	- Re-analyze the initial calibration standard and/or evaluate/correct instrument malfunction to obtain initial calibration and continuing calibration check standards that meet criteria.
Initial Calibration Verification Standard (ICV)	Initially analyze a ICV immediately following an initial calibration.	- %D for the ICV must within ±20% of the true value. - Retention time of all quantitated peaks must be within RT	- If the reason for the failure of the ICV appears to be a poor injection (or a degraded standard solution), the ICV will be re-injected (or re-prepared and re-injected) immediately following the failed ICV. If upon re-injection, the ICV meets all the acceptance criteria and there is no apparent impact on the sample data the analytical sequence will continue and samples will not be reanalyzed. The associated sample data will be reported.

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Continuing Calibration Verification Standard (CCV)	- After the initial CCV of the sequence, a CCV must be analyzed after 10 samples. - Analytical sequence must end with analysis of a CCV. - CCV must be analyzed at least once per 12 hour analytical shift.	- %D for the CCV must be within ±20% of the true value. - Retention time of all quantitated peaks must be within RT - All samples must be bracketed by CCV that meet all criteria stated above.	- If the reason for the failure of the CCV appears to be a poor injection (or a degraded standard solution), the CCV will be re-injected (or re-prepared and re-injected) immediately following the failed CCV. If upon re-injection, the CCV meets all the acceptance criteria and there is no apparent impact on the sample data the analytical sequence will continue and samples will not be reanalyzed. The associated sample data will be reported. - -If acceptable CCVs are observed later in the sequence, samples bracketed by acceptable CCVs will be reported. Samples between the failed CCV and prior/ subsequent complaint CCV will be re-analyzed. - Exception: Samples that are non-detect for analytes of interest may be reported with a high bias if a bracketing CCV fails high.
-Retention Time (RT)	- RT windows are established annually. -Each sample analysis: Rely on RT windows to identify PCB Aroclor to report. Also use pattern recognition and professional judgment for peaks that shift from RT windows, because compound composition may shift RT for GC peaks.	- Each quantitated peak and surrogate peak should be within established windows.	-Inspect chromatographic system for malfunction, correct problem. Perform re-analysis or re-calibration if necessary.

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Method Blank	-One per extraction batch of ≤20 samples of the same matrix per day. -Should be analyzed with other associated batch QC samples on the same instrument, but not all samples. -Must undergo all sample preparative procedures.	- Concentration does not exceed the RL/MDL for any PCB Aroclor. - Percent recovery for surrogates must be within method limits of 60-140%. Only one surrogate analyte needs to meet established control limits for the analysis to be valid	- Re-analyze method blank to determine if instrument contamination was the cause. If method blank re-analysis passes, then report samples. -If method blank is found to contain PCB contamination above the RL/MDL for any PCB Aroclor compound, then re-extract and re-analyze all associated samples. -If no sample is available for re-extraction, report data with a B-flag to indicate method blank contamination.
Laboratory Control Spike (LCS)	- One per extraction batch of ≤20 samples per matrix per day. - Should be analyzed with other associated batch QC samples on the same instrument, but not all samples.	-Percent recovery must be within method limits of 70-130%.(spiked with Aroclor 1242) - Percent recovery for surrogates must be within method limits of 60-140%. Only one surrogate analyte needs to meet established control limits for the analysis to be valid	-Re-analyze LCS to determine if instrument was the cause. If LCS passes, then report samples. -If LCS recovery is still out of limits, then re-extract and re-analyze all associated samples. -If no sample is available for re-extraction, report data flagged to indicate LCS failed recovery.
Laboratory Control Sample Duplicate (LCSD)	- One per extraction batch of ≤20 samples per day. - Should be analyzed with other associated batch QC samples on the same instrument, but not all samples.	-Percent recovery must be within method limits of 70-130%.(spiked with Aroclor 1242) - Relative percent difference (RPD) should be within 30% - Percent recovery for surrogates must be within method limits of 60-140%. Only one surrogate analyte needs to meet established control limits for the analysis to be valid	-Re-analyze LCSD to determine if instrument was the cause. If LCSD passes, then report samples. -If LCSD recovery is still out of limits, the re-extract and re-analyze all associated samples. -If no sample is available for re-extraction, report data flagged to indicate LCSD failed recovery.

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	-One MS/MSD per extraction batch of ≤20 samples of similar matrix. - An MS/DUP may be appropriate in place of MS/MSD in matrices where detectable amounts of analytes are known to be present.	-Percent recovery must be within method limits of 70-130%.(spiked with Aroclor 1242) -Relative percent difference (RPD) should be within 30%. - Percent recovery for surrogates must be within method limits of 60-140%. Only one surrogate analyte needs to meet established control limits for the analysis to be valid	-Re-analyze MS and/or MSD to determine if instrument was the cause. If MS and/or MSD pass, then report samples. -Check for errors such as calculations and spike preparation. -Check parent sample results and surrogate recovery for indications of matrix effects. -If no errors are found, and the associated LCS is within limits, then sample matrix effects are likely the cause. Note exceedance in case narrative.
Sample Duplicate (DUP)	- When used in place of an MS/MSD: One MS/DUP per extraction batch of ≤20 samples of similar matrix.	-Relative percent difference (RPD) should be within 30%. - Percent recovery for surrogates must be within method limits of 60-140%. Only one surrogate analyte needs to meet established control limits for the analysis to be valid outside of criteria)	- Re-analyze the sample and sample duplicate to determine if the instrument was the cause. If RPD is within limits in re-analysis, then report the data. - Check surrogate recovery for indications of matrix effects. - Check for calculation errors. - If no errors are found, and the associated LCS is within limits, than sample matrix effects are likely the cause. Note exceedance in the case narrative.

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Surrogates	-Calibrated as target compound in the Aroclor 1660 standards. -Surrogates are added to all calibration check standards, blanks, samples and QC samples.	- Percent recovery for surrogates are 60-140%. Only one surrogate analyte needs to meet established control limits for the analysis to be valid.	-Re-analyze the affected sample or QC sample to determine if instrument was the cause. If surrogate passes, then report samples. -Check for errors in surrogate calculation and surrogate solutions. -If no problem is found, then re-extract and re-analyze the sample. -If the re-extraction produces surrogate recovery still out of limits, then report data flagged to indicate surrogate failed recovery, confirmed by re-extraction and analysis. -If no sample exists for re-extraction, report data flagged to indicate surrogate failed recovery or have a client re-sample.

14. Data Analysis and Calculations

14.1. Percent Recovery

$$\mathrm{P}\,(\%) = \frac{(A)}{(T)} X 100\%$$

P = Percent recovery, %
A = concentration of analyte (PCB) in the spike sample aliquot
T = Know true values of the spike concentration

14.2. RPD (Relative Percent Difference)

$$\%RPD = \left(\frac{Dup1 - Dup2}{Ave} \right) x 100$$

RPD = Relative Percent Difference
DUP1 = The greater of the measured values
DUP2 = The lesser of the measured values
AVG = Average of the two analyses

14.3. PCB Solution concentration calculation from initial Calibration by Average Response Factor:

$$\text{Concentration } (\mu\text{g/mL}) = \frac{(X)}{(Y)}$$

X = Area response of PCB quant peak
Y = Average Response Factor from Calibration

14.4. Capillary GC: Sample calculations:

14.4.1. The concentration of each identified PCB Aroclor in a sample will be calculated based on the sample weight or volume.

14.4.2. Biota Tissue:

$$\text{Concentration } (\mu\text{g/Kg}) = \frac{(C_x)(DF)(U_f)(V_t)}{(V_i)(W_s)}$$

Where:
C_x = On-column concentration in extract (µg/mL).
DF = Dilution factor.
U_f = Correction factor.
V_t = Volume of final extract (µL).
V_i = Volume injected (µL).
W_s = Weight of sample extracted (g).

14.5. The calculated concentration for each PCB Aroclor will be compared to its respective sample-specific reporting limit (RL) and method detection limit (MDL). The results with concentrations at or above the MDL but below RL will be reported as detects and flagged as estimated (J-flag). The results for peaks with concentrations at or above the RL would be reported as unqualified numeric values.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. The GC analyst is responsible for generating the data and also is the initial individual to review the data. This would include inspection of the chromatographic data, processing the raw data, producing all required data forms, inspection of calibration curves for compliance, surrogate recovery, laboratory control spike recovery, matrix spike/matrix spike duplicate recovery, and continuing calibration compliance.

15.2. Once the initial review of the data is performed by the analyst, decisions are made at that time to accept the data if all criteria are met or to reject sample data if any of the quality control parameters or limits are out of control. Depending on the situation, samples requiring re-extraction will be notified to the appropriate extraction personnel, sample extracts requiring re-injection will be queued for analysis, new calibrations may have to be performed, or samples re-analyzed due to failing continuing check standards.

15.3. The analyst may also consult with the Quality Manager as to the best form of action to take or if the situation warrants corrective action beyond routine practices. If no recourse is available and the data is to be reported out of criteria, a Case Narrative Report is generated and the deviation is documented and reported to the client. The Case Narrative Report is filed with the data and is also useful for production of case narratives that are issued with the final data reports. If a problem exists that requires follow-up to rectify, a LabTrack Ticket (LTT) is issued to document the problem found, steps taken to resolve the problem, and what samples were affected. This LTT is reviewed by the Quality Manager and lab management to verify that appropriate actions have been taken to correct the problem.

15.4. Please see Table 13. for specific Quality Assurance Acceptance Criteria.

16. Corrective Actions for Out-of-Control Data

16.1. See table in section 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Data that is detected to be out-of-control for any reason, when compared to method acceptance criteria, will addressed in the following manner:

17.1.1. If the problem exists with the gas chromatographic instrumentation, appropriate action will be taken to repair and perform maintenance to bring the instrument back to operation condition. Once the instrumentation is determined to be correctly operating analysis can begin again.

17.1.2. If the problem exists with calibration standard solutions, the analyst will prepare new standards and discard the standard solutions that are suspect. Instrument calibration can be performed and analysis can begin once system is control.

17.1.3. If the problem exists with sample extraction and extract preparation, the extraction step that is producing the out of-control situation will be diagnosed and rectified. Once the troubleshooting procedures correct the problem extraction can once again occur and analysis can continue.

17.1.4. In situations where data is reported under out-of-control conditions, the data will be annotated with data qualifiers and/or appropriate descriptive comments defining the nature of the excursion in the sample case narrative. If warranted, a LabTrack Ticket will be issued to define the problem, steps to correct the problem, and final resolution.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. **Method Detection Limit (MDL) Study**: Method detection limits are established according to the method established in 40 CFR 136, Appendix B. See SOP S-GB-Q-020 Determination of LOD and LOQ current revision for details.

18.3. **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-ALL-Q-020, Training Procedures.

19. Method Modifications

19.1. Not applicable to this SOP.

20. Instrument/Equipment Maintenance

20.1. Not applicable to this SOP.

21. Troubleshooting

21.1. Not applicable to this SOP.

22. Safety

22.1. Safety glasses and disposable gloves must be worn when handling samples and extracts.

22.2. All manipulations of sample extracts should be conducted inside a chemical fume hood. Manipulation of sample extracts outside of a fume hood should be minimized by the analyst.

22.3. Safe laboratory practices should be followed by the analyst at all times when conducting work in the lab. The analyst should refer to the reference file of material safety data sheets to familiarize themselves with the precautions for handling solvents and chemicals used to process samples. The analyst should refer to the laboratory chemical hygiene plan for further safety information.

22.4. Samples remaining after analysis should either be returned to the customer for disposal or disposed of through the laboratory's disposal plan. Refer to the sample custodian for assistance and also SOP S-GB-W-001, disposal of laboratory waste.

23. Waste Management

23.1. All applicable federal and state rules and regulations governing hazardous waste will be followed when disposing of laboratory waste generated during the execution of this method.

23.2. Please refer to SOP S-GB-W-001 regarding how hazardous waste is handled and disposed of by the laboratory.

24. Pollution Prevention

24.1. Pollution prevention is practiced in the laboratory by minimizing usage of solvents and chemicals, so that disposal of waste generated is held to the smallest amount possible. This is directly linked to the types of extraction procedures in place at the laboratory to reduce the volumes of solvents used for semi-volatile extraction procedures.

24.2. The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.4. U.S. EPA SW-846 Method 8082A "Test Methods for Evaluating Solid waste; Volume 1B Laboratory Manual Physical/Chemical Methods", Office of Solid Waste and Emergency Response, Third Edition, Final Update III, December 1996.

25.5. U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures of the Analysis of Pollutants", July, 1988.

25.6. New York State Department of Health, "Environmental Laboratory Approval Program Certification Manual", Wadsworth Center for laboratories and Research, 1996.

25.7. Guide to Environmental Analytical Methods", third edition, Genium Publishing Corporation, 1997.

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I: GC Operating Parameters

26.2. Attachment II: Chromatograms.

27. Revisions

Document Number	Reason for Change	Date
S-GB-O-067-Rev.00	Used Pace – Schenectady Laboratory SOP S-NY-O-314 to create local SOP.	14Jul2017
S-GB-O-067-Rev.01	Cover page: Updated Supersedes Section to: S-NY-O-314-Rev.03 (Appendix A3-1 of 2016 RAM QAPP Attachment A). Section 5.1: Removed language "and are subject to change without update to this SOP". Section 5.1 Table: Changed Matrix from biota to fish	29Nov2017

Attachment I: Example GC Operating Parameters

INSTRUMENT CONTROL PARAMETERS: 40GCSVJ

C:\msdchem\1\METHODS\GEBIOTA2017.M
Fri Jul 14 11:12:45 2017

Control Information

Sample Inlet : GC
Injection Source : GC ALS

No Sample Prep method has been assigned to this method.

6890 GC METHOD

OVEN
Initial temp: 150 'C (On)
Initial time: 1.41 min
Ramps:

#	Rate	Final temp	Final time
1	17.50	300	3.00
2	0.0(Off)		

Post temp: 0 'C
Post time: 0.00 min
Run time: 12.98 min
Maximum temp: 360 'C
Equilibration time: 0.50 min

FRONT INLET (SPLIT/SPLITLESS)
Mode: Split
Initial temp: 250 'C (On)
Pressure: 29.94 psi (On)
Split ratio: 15:1
Split flow: 30.1 mL/min
Total flow: 39.1 mL/min
Gas saver: Off
Gas type: Hydrogen

BACK INLET (SPLIT/SPLITLESS)
Mode: Split
Initial temp: 250 'C (On)
Pressure: 29.94 psi (On)
Split ratio: 15:1
Split flow: 30.1 mL/min
Total flow: 38.6 mL/min
Gas saver: Off
Gas type: Hydrogen

COLUMN 1
Capillary Column
Model Number: Phenomen ZB-1MS
20m x 0.18mm x 0.18um
Max temperature: 360 'C
Nominal length: 20.0 m
Nominal diameter: 180.00 um
Nominal film thickness: 0.18 um
Mode: constant flow
Initial flow: 2.0 mL/min
Nominal init pressure: 29.95 psi
Average velocity: 86 cm/sec
Inlet: Front Inlet
Outlet: Front Detector
Outlet pressure: ambient

COLUMN 2
Capillary Column
Model Number: Phenomen ZB-1MS
20m x 0.18mm x 0.18um
Max temperature: 360 'C
Nominal length: 20.0 m
Nominal diameter: 180.00 um
Nominal film thickness: 0.18 um
Mode: constant flow
Initial flow: 2.0 mL/min
Nominal init pressure: 29.95 psi
Average velocity: 86 cm/sec
Inlet: Back Inlet
Outlet: Back Detector
Outlet pressure: ambient

FRONT DETECTOR (µECD)
Temperature: 300 'C (On)
Mode: Constant makeup flow
Makeup flow: 35.0 mL/min (On)
Makeup Gas Type: Nitrogen
Electrometer: On

BACK DETECTOR (µECD)
Temperature: 300 'C (On)
Mode: Constant makeup flow
Makeup flow: 35.0 mL/min (On)
Makeup Gas Type: Nitrogen
Electrometer: On

SIGNAL 1
Data rate: 50 Hz
Type: front detector
Save Data: On

SIGNAL 2
Data rate: 50 Hz
Type: back detector
Save Data: On

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Attachment I: Example GC Operating Parameters (continued)

```
   Start Save Time:  1.00 min
   Stop Save Time:  10.00 min
   Zero:  0.0 (Off)
   Range:  0
   Fast Peaks:  Off
   Attenuation:  0

COLUMN COMP 1
   Derive from front detector

   Start Save Time:  1.00 min
   Stop Save Time:  10.00 min
   Zero:  0.0 (Off)
   Range:  0
   Fast Peaks:  Off
   Attenuation:  0

COLUMN COMP 2
   Derive from back detector

POST RUN
   Post Time: 0.00 min

TIME TABLE
   Time        Specifier                    Parameter & Setpoint

                              GC Injector

   Front Injector:
      Sample Washes                  1
      Sample Pumps                   3
      Injection Volume            1.00 microliters
      Syringe Size                10.0 microliters
      PreInj Solvent A Washes        2
      PreInj Solvent B Washes        1
      PostInj Solvent A Washes       2
      PostInj Solvent B Washes       1
      Viscosity Delay                0 seconds
      Plunger Speed               Fast
      PreInjection Dwell          0.00 minutes
      PostInjection Dwell         0.00 minutes

   Back Injector:
      Sample Washes                  1
      Sample Pumps                   3
      Injection Volume            1.00 microliters
      Syringe Size                10.0 microliters
      PreInj Solvent A Washes        2
      PreInj Solvent B Washes        1
      PostInj Solvent A Washes       2
      PostInj Solvent B Washes       1
      Viscosity Delay                0 seconds
      Plunger Speed               Fast
      PreInjection Dwell          0.00 minutes
      PostInjection Dwell         0.00 minutes

 Column 1 Inventory Number : GCSJ1
 Column 2 Inventory Number : GCSJ1

PostRun InstCntl macro(s) exist: msacq2.mac

                    END OF INSTRUMENT CONTROL PARAMETERS
                    ------------------------------------

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```

Attachment II: Chromatograms (ZB-1MS Column/Hydrogen Carrier Gas)

FIGURE 1: A1016 at 0.500ug/mL Plot

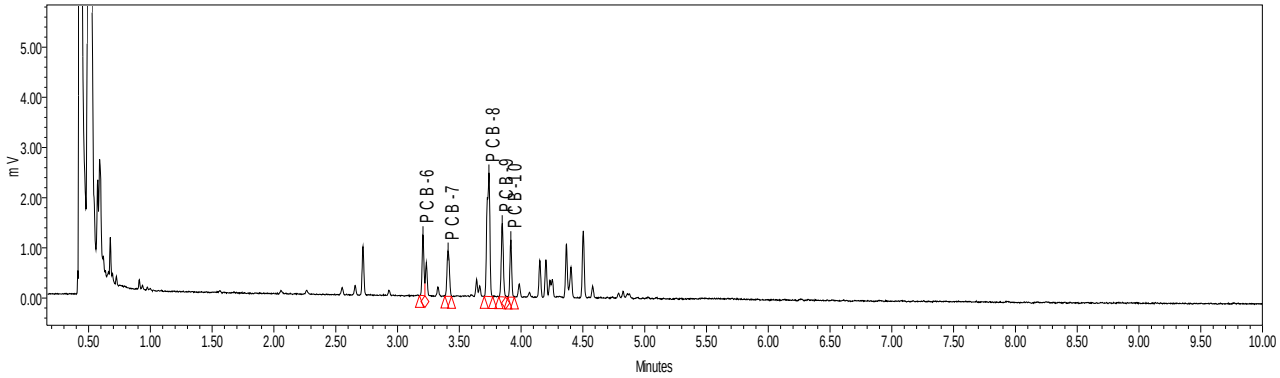


FIGURE 2: A1221 at 0.500ug/mL Plot

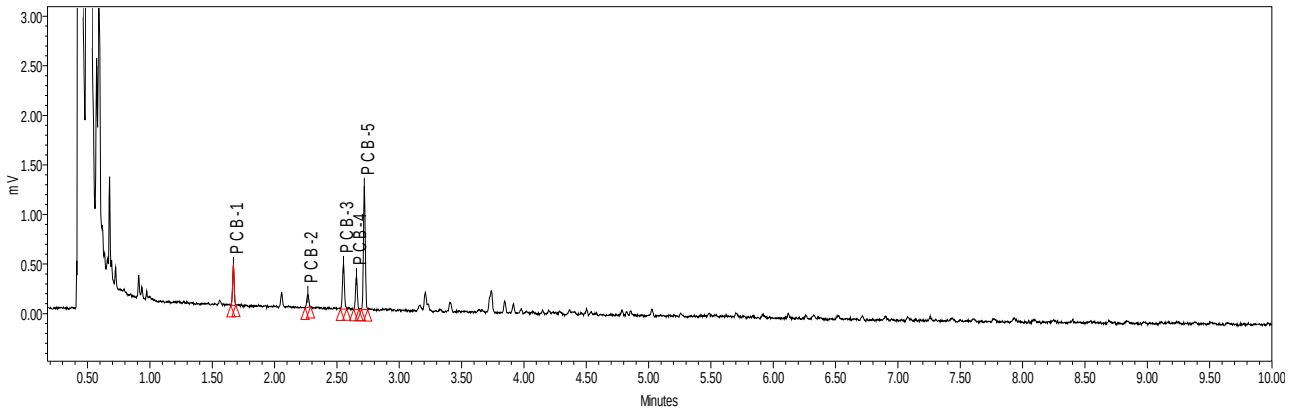
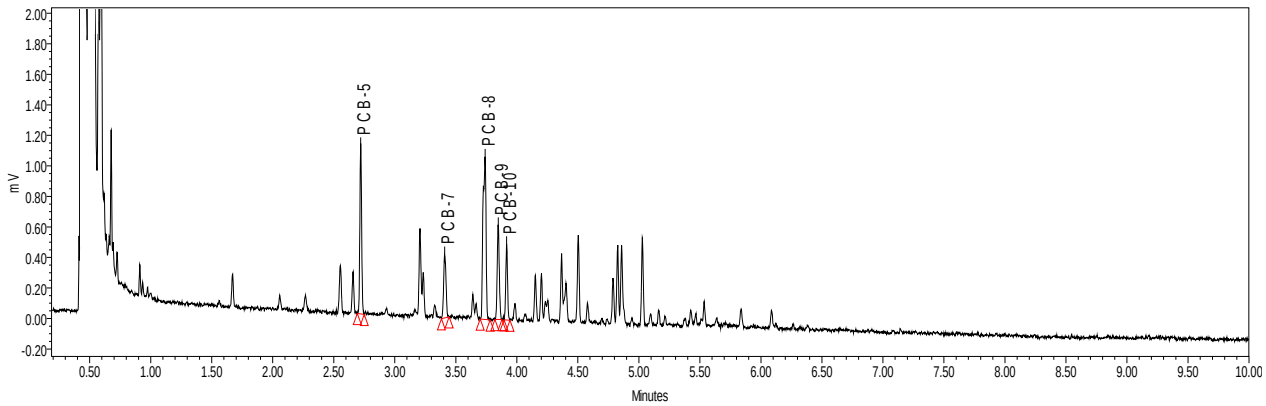


FIGURE 3: A1232 at 0.500ug/mL Plot



Attachment II: Chromatograms (ZB-1MS Column/Hydrogen Carrier Gas) (continued)

FIGURE 4: A1242 at 0.500ug/mL Plot

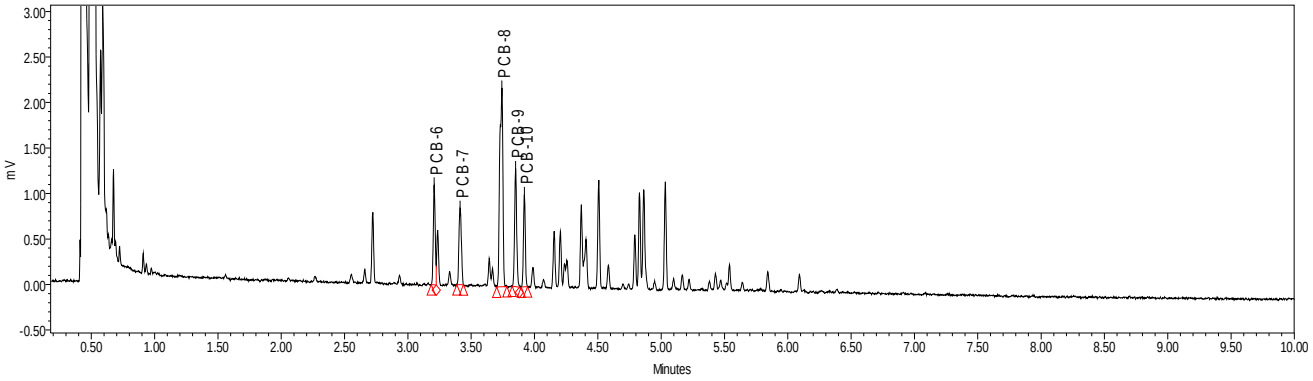


FIGURE 5: A1248 at 0.500ug/mL Plot

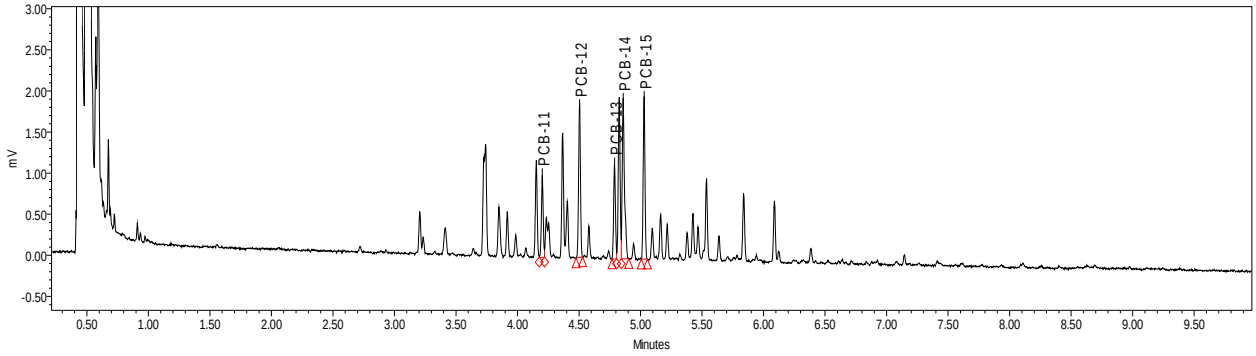
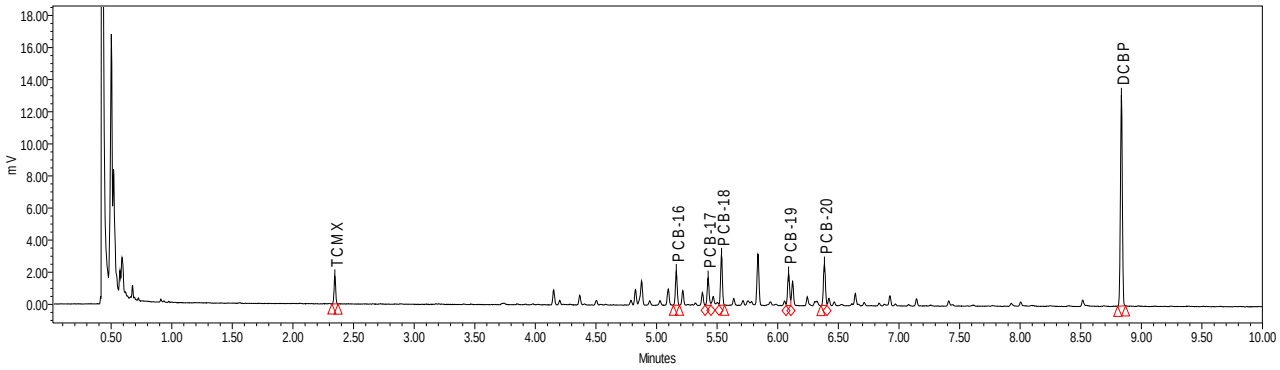


FIGURE 6: A1254 at 0.500ug/mL w/ TCMX & DCBP at 10/100ug/L Plot



Attachment II: Chromatograms (ZB-1MS Column/Hydrogen Carrier Gas) (continued)

FIGURE 7: A1260 at 0.500ug/mL Plot

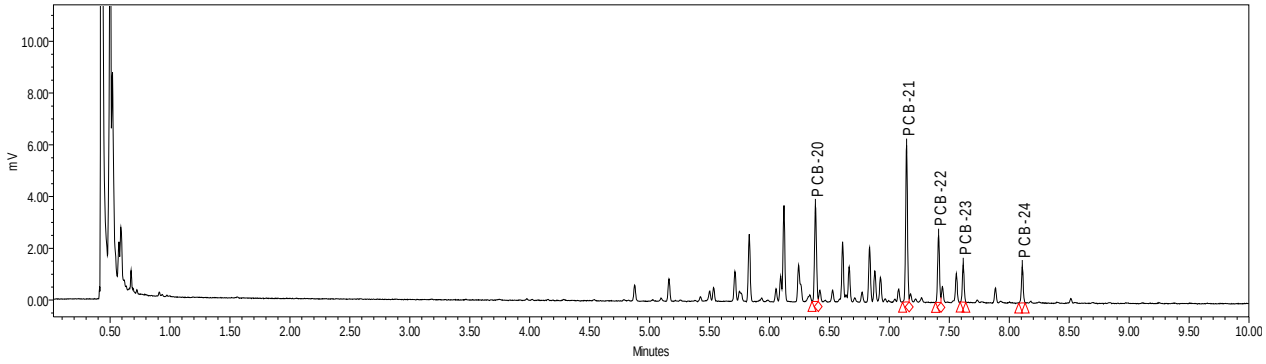


FIGURE 8: A1262 at 0.500ug/mL Plot

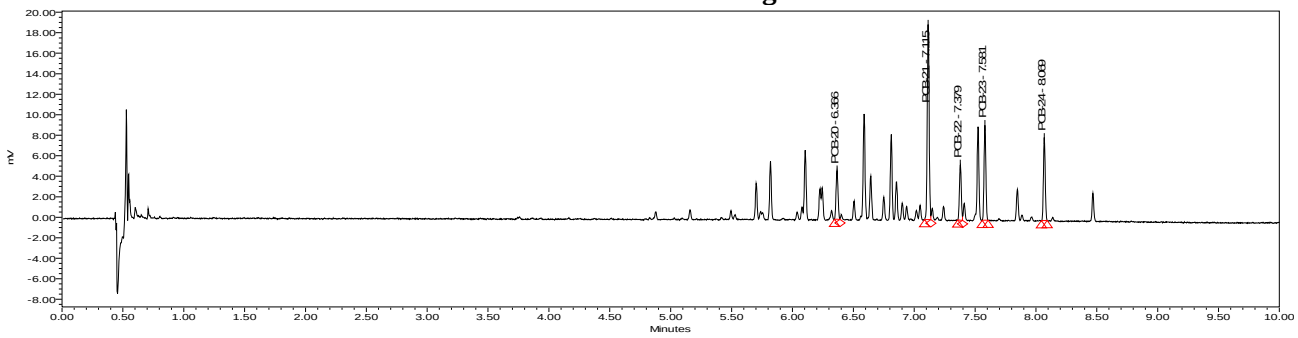
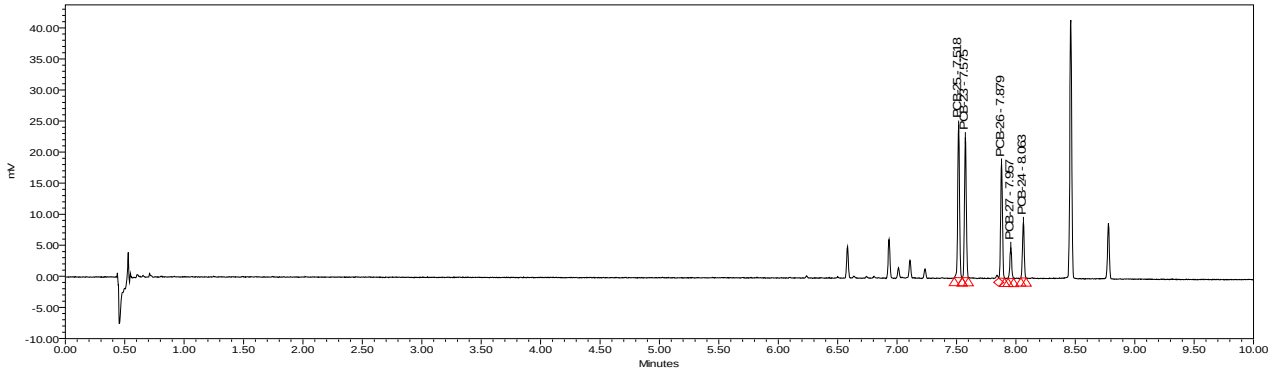


FIGURE 9: A1268 at 0.500ug/mL Plot



United States Environmental Protection Agency - Region 2
HUDSON RIVER OFFICE
187 Wolf Road, Suite 303, Albany, New York 12205
Telephone/Fax: 518/407-0400 • Email: EPAHRFO@outlook.com

April 23, 2019

Mr. Robert Gibson
Senior Project Manager – Environmental Remediation
General Electric Company
1 River Road
Bldg. 5-7W
Schenectady, New York 12345

Re: Corrective Action Memo #15 to the Phase 2 Remedial Action Monitoring Project Quality Assurance Project Plan – Use of Standard Reference Material (SRM) provided by National Institute of Standards and Technologies (NIST)

Dear Mr. Gibson:

As a follow up to EPAs approval of CAM15 (revised on February 7, 2018), this letter provides further clarification regarding the need for the use of reference material and congener analytical methods for the 2018 Hudson River fish that have been collected. Information outlined below includes consideration of ongoing discussions between EPA and GE in coordination with NYSDEC. Incorporation of a NIST SRM into the OM&M analytical program is important for the long-term consistency in quantifying PCB concentrations in fish which will need to be compared over many years as the river continues to recover. The importance of the NIST SRM becomes even more relevant as the absolute magnitude of changes in PCB concentration decrease in future years making the ability to detect changes over time more sensitive to year to year variability. GE can begin 2018 fish processing and analysis with the following understanding:

Analytical Program:

- A minimum of 5% of fish will be analyzed using EPA M1668. The M1668 samples should be evenly distributed across species and locations.
- Seven samples of NIST SRM 1946 (Lake Superior PCB Congener Fish Tissue) and NIST SRM 1947 (Lake Michigan PCB Congener Certified Fish Tissue) will be run by M8082 separately from the Hudson River Fish samples to develop data quality indices. This analysis is necessary because NIST SRM 1946 and NIST SRM 1947 are not certified for total PCBs by Aroclor M8082 (the primary method used by EPA to evaluate Hudson River project fish). These NIST standards are certified for specific PCB congeners. The results from this analysis will be evaluated to confirm that NIST SRM 1946 and NIST SRM1947 are acceptable to EPA for use in the analytical program. The data quality indices developed from this analysis will be incorporated into the OM&M fish sampling program beginning with the 2018 fish. It is anticipated that the two SRMs will have different PCBs patterns therefore providing a wider range of PCBs to consistently reference over time. In the event the data quality indices are not fully established, before it is necessary to run the LHR fish due to hold times, GE should contact EPA to discuss next steps.
- NIST SRM material will be run at a rate of 1(NIST SRM):40 fish tissue samples for both lipid and PCBs. The use of the two SRMs (NIST SRM 1946 and NIST SRM 1947, if both are

appropriate for use) should be alternated throughout the M8082 analytical program such that equal number of each are analyzed. For M1668, a minimum of one NIST SRM 1946 and NIST SRM 1947 will need to be analyzed for a calendar year of fish collection.

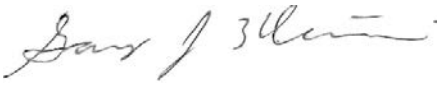
- Duplicates of fish homogenate will continue to be run at 1:20. Matrix Spikes will continue to be run at 1:20 until data quality indices are established and the NIST SRM material is confirmed acceptable to EPA (at which time NIST SRM can take the place of the matrix spikes in a particular SDG, such that either a matrix spike or NIST SRM are run at a rate of 1:20).

Other Items:

- GE will provide updated PACE standard operating procedures (SOPs) to reflect observations made during laboratory audits last year. These updates will be consistent with past practices on the project.
- GE will provide SOPs for analysis of fish tissue and NIST SRM samples using M1668.
- EPA and GE will continue to coordinate on the re-analysis of select 2016 fish that have been analyzed at the Pace Schenectady lab.
- As previously agreed, GE will proceed with the collection of fish in Reaches 1 – 4. This work should be conducted as outlined in the proposed approach for fish sampling in reaches 1-4 provided to GE on 9/6/2018 and attached for reference. GE will attempt to collect equal numbers of fish of each species from the primary and secondary transects identified by GE. If the target number of fish cannot be collected from the identified transect using a similar level of effort in other river reaches, GE and EPA will discuss next steps as necessary.

Please coordinate with EPA on the schedule for fish processing. As in past years, EPA will have project staff oversee fish processing.

Sincerely,



Gary Klawinski, Project Director
USEPA Hudson River Office

ENVIRONMENTAL® STANDARDS

Setting the Standards for Innovative Environmental Solutions

TECHNICAL MEMORANDUM

Date: August 16, 2019

To: Bob Gibson – General Electric Company

From: David Blye – Environmental Standards, Inc.
Meg Michell – Environmental Standards, Inc.
Jared Acker – Environmental Standards, Inc.

Copy: Jim Ryan – Anchor QEA
Mark Meyers – Anchor QEA
Zheng Wang – Anchor QEA
File

Subject: Pace GB 2017 fish matrix spike evaluation

BACKGROUND

Prior to 2017, PCB Aroclor fish analyses took place at Pace Analytical Services (Pace) Schenectady, New York. Pace ceased laboratory operations at Schenectady, New York, at the end of 2016 and fish analyses were transferred to the Pace Green Bay, Wisconsin (Pace GB) laboratory. As required by the Phase 2 Hudson River Remedial Action Monitoring Quality Assurance Project Plan (Phase 2 RAM QAPP), matrix spikes (MS) and laboratory duplicates (LD) are analyzed at the rate of one pair per sample batch (up to 20 samples) for fish samples. In 2017, all MS samples were spiked with Aroclor 1242 in accordance with standard operating procedures (SOPs) extraction by SW-846 Method 3541 (SOP S-GB-O-068-rev.01) and the analysis for PCB Aroclors using SW-846 Method 8082A (SOP S-GB-O-067-REV.01) included as Attachments 2 and 3, respectively, of the Hudson River Phase 2 Remedial Action Monitoring Program 2017 Corrective Action Memorandum No. 15 (CAM 15, Feb 2018) to the Phase 2 RAM QAPP.

Although Aroclor 1242 is spiked into the MS due to its historical use at the site, the Aroclor pattern observed in Hudson River fish has been changing throughout the years by weathering and various biological transformations. Currently, a combination of Aroclors 1221, 1248, 1254, and/or 1260 are typically reported in the Hudson River fish samples in order to provide a more accurate quantitation of the altered Aroclor pattern. Many of the quantitation peaks for each of the target Aroclors (including Aroclor 1242) are present in the majority of sample chromatograms such that each target Aroclor often quantitates to a concentration above the method detection limit (MDL). The Aroclors quantitated in each sample are selected by laboratory personnel to provide the best estimate of the total PCB concentration.

The laboratory information management system (LIMS) used at Pace Schenectady was able to subtract parent sample background (Aroclor 1242) by a manual process even when Aroclor 1242 was not reported in the parent sample. Although this was an atypical laboratory practice, it resulted in more accurate MS recovery quantitation as it accounted for PCB background that otherwise elevated the MS recovery. Due to limitations in the software used to report PCB Aroclor data at Pace GB, parent sample background of a non-reported Aroclor cannot be subtracted from MS results in order to calculate MS recovery. During validation of the 2017 fish data, it was observed that nearly all MS results were biased high due to the limitation.

In addition, due to limitations in the software used to report PCB Aroclor data at Pace GB, Pace GB could not limit the EDD to only report the spiked target analyte (Aroclor 1242) and the surrogates as was done by Pace Schenectady in the past. Due to this reporting difference, the data verification module (DVM) did not evaluate whether the background was greater than 4× the spike amount correctly (each Aroclor concentration in the parent sample was compared to the spike amount rather than only the spiked analyte, Aroclor 1242). MS recovery is not evaluated when the background is greater than 4× the spike amount.

Due to the reporting differences at Pace GB from Pace Schenectady, MS qualification was improperly applied by the current DVM process.

RESPONSE

In order to obtain more accurate MS recoveries, it was determined that all 2017 MS parent samples would be manually quantitated for the “Aroclor 1242” background by Environmental Standards, Inc. (Environmental Standards). These calculations were based off the peak area counts, average response factors from calibrations, dilution factors, correction factors, final volumes, injection volumes, and sample weights obtained from the full data packages. The calculations used to generate Aroclor 1242 results in each MS parent sample were obtained from Sections 14.3 and 14.4 of the Pace GB SOP S-GB-O-067-REV.00. In nearly all samples, all Aroclor 1242 peaks were integrated and had peak area amounts. In cases were a peak was missed, or no peak was present, 0 was used for the peak area in the calculation. Due to small sample count or split samples from some sample delivery groups (SDGs), not every SDG had its own MS. The table below represents a summary of all 2017 MS results. The “Recalculated Background Result (µg/kg)” column displays the manually quantitated background result, and the “Recalculated MS %R” column displays the corrected MS recovery.

SDG	Parent Sample	Spike Added (µg/kg)	MS Result (µg/kg)	Recalculated Background Result (µg/kg)	Original MS %R	Recalculated MS %R	Criteria (70-130%) Met?
40148598	TZ1-170418-01-STB-01	250	313	57	125%	102%	Yes
40148722	TZ1-170420-01-STB-12	250	235	11	94%	90%	Yes
40150291	CS1-170518-01-YP-03	471	531	36	113%	105%	Yes
40150294	CS1-170518-01-BB-06	250	266	27	107%	96%	Yes
40150295	CS1-170518-01-STB-02	250	519	295	208%	90%	Yes
40150340	AT1-170519-01-STB-10	250	281	26	112%	102%	Yes
40151542	TD3-170612-01-SMB-03	249	340	115	136%	90%	Yes
40151552	TD5-170612-01-BB-01	250	354	99	142%	102%	Yes

SDG	Parent Sample	Spike Added (µg/kg)	MS Result (µg/kg)	Recalculated Background Result (µg/kg)	Original MS %R	Recalculated MS %R	Criteria (70-130%) Met?
40151605	TD2-170612-01-LMB-01	250	263	17	105%	98%	Yes
40151612	FD1-170613-01-YP-04	250	242	3	97%	96%	Yes
40151613	FD1-170613-01-SMB-03	251	238	0	95%	95%	Yes
40151616	ND1-170613-01-BB-01	251	NA	NA	NA	NA	NA[1]
40151619	SW1-170613-01-YP-04	251	308	50	123%	103%	Yes
40151705	FD1-170613-01-BB-02	250	254	1	101%	101%	Yes
40157487	SW3-170614-01-LMB-04	250	313	77	125%	94%	Yes
40157488	SW2-170614-01-SMB-01	251	445	173	178%	108%	Yes
40157488	SW2-170614-01-YP-01	250	284	45	113%	96%	Yes
40157489	SW3-170614-01-BB-05	249	352	109	141%	98%	Yes
40157492	SW4-170614-01-BB-02	250	280	45	112%	94%	Yes
40157694	ND3-170615-01-YP-03	250	453	176	181%	111%	Yes
40157698	ND5-170615-01-LMB-01	250	289	46	116%	97%	Yes
40157703	ND3-170615-01-BB-02	249	465	239	186%	91%	Yes
40157822	AT1-170616-01-SMB-04	249	281	30	113%	101%	Yes
40157828	AT1-170616-01-CHC-03	250	421	148	168%	109%	Yes
40157832	AT1-170616-01-YP-03	250	312	59	125%	101%	Yes
40157932	CS1-170620-01-YP-08	251	328	56	131%	108%	Yes
40157935	CS1-170620-01-WP-01	251	272	19	108%	101%	Yes
40157937	TD1-170828-01-GOSH-01	249	526	159	211%	147%	No[2]
40157940	ND2-170828-01-GOSH-02	250	1220	1060	487%	64%	NA[3]
40157948	TD2-170828-01-SPSH-01	460	649	153	141%	108%	Yes
40158010	FD1-170829-01-PKSD-01	500	469	4	94%	93%	Yes
40158013	SW2-170829-01-PKSD-01	306	1360	1053	445%	100%	Yes
40158017	FD1-170829-01-MMSH-05	705	709	5	101%	100%	Yes
40158023	FD1-170829-01-PKSD-11	590	650	79	110%	97%	Yes
40158025	AT1-170830-01-PKSD-01	250	334	36	134%	119%	Yes
40158028	SW4-170830-01-PKSD-01	420	835	317	199%	123%	Yes
40158029	AT1-170830-01-SFSH-02	359	514	92	143%	118%	Yes

1 - Recovery is not applicable because the MS analysis was performed at a 40-fold dilution.

2 - A low MS recovery (>130%) was obtained. Positive Aroclor results should be flagged estimated ("J").

3 - Recovery is not applicable because the background was >4× the spike amount.

CONCLUSIONS

Overall, acceptable MS recoveries were observed for the majority of 2017 fish data with one exception. The qualification based on the MS evaluation originally performed by DVM for the 2017 data set has been removed from the database. In addition, qualification has been applied to sample TD1-170828-01-GOSH-01 based on the MS evaluation shown above.

The Aroclor spiked in the MS sample was changed from Aroclor 1242 to Aroclor 1248 starting with 2018 fish samples, which allows the laboratory to background subtract without the need for manual intervention and is better represent the altered Aroclor currently observed in fish.

The fish database is being migrated to a new EQuIS-based database. The MS evaluation for the DVM process will be evaluated as part of this migration.

ENVIRONMENTAL STANDARDS
Setting the Standards for Innovative Environmental Solutions

TECHNICAL MEMORANDUM

Date: August 19, 2019

To: Bob Gibson – General Electric Company

From: David Blye – Environmental Standards, Inc.
Meg Michell – Environmental Standards, Inc.

Copy: Mark LaRue – Anchor QEA
Jennifer Benaman – Anchor QEA
Chris Yates – Anchor QEA
Mark Meyers – Anchor QEA
Sheng Wang – Anchor QEA
File

Subject: Results of Re-Extraction of Samples OWS-THIS-T170726143612

BACKGROUND

As required by the Phase 2 Hudson River Remedial Action Monitoring Quality Assurance Project Plan (Phase 2 RAM QAPP), weekly far-field water column samples are collected for the Remedial Action Monitoring Program (RAMP) during non-dredging periods. Thompson Island Dam (TID) sample OWS-THIS-T170726143612 (collected July 26, 2017) was submitted to Vista Analytical Laboratory (Vista) for congener PCB analysis by EPA 1668C and to Pace Analytical in Minneapolis, Minnesota (Pace) for TSS analysis. The sample was grouped with other samples in Vista sample delivery group (SDG) 1700951 and Pace SDG 10397544. Vista reported a total PCB concentration of 46.5 ng/L and Pace reported a TSS concentration of 68.7 mg/L in the sample, which were inconsistent with the results observed at the TID during similar timeframes (refer to Table 1; the results presented reflect the Total PCB results after electronic data verification was performed).

Table 1. Results for TID Samples Collected Near July 26, 2017

Sample Identification	Sample Type	Collection Date and Time	Total PCB (ng/L)	TSS (mg/L)
OWS-THIS-T170713130313	ENV	7/13/2017 11:31	5.39	7.7
OWS-BDUP-T170713130336	DUP	7/13/2017 11:31	5.43	7.3
OWS-THIS-T170720143207	ENV	7/20/2017 12:06	7.16	2.6
OWS-THIS-T170726143612	ENV	7/26/2017 11:52	46.5	68.7
OWS-THIS-T170801141823	ENV	8/1/2017 12:45	8.14	2.1
OWS-THIS-T170808141929	ENV	8/8/2017 12:48	7.22	4.6

RESPONSE

The PCB results reported by Vista for sample OWS-THIS-T170726143612 were from a re-extraction. Vista performed this reextraction because of minor method blank contamination. Therefore, the reported results (from the reextraction) were compared to the results of the original extraction. The TID results for the original extraction and analysis and results for the reextraction and reanalysis, are provided in Table 2 (the results presented reflect the Total PCB results after electronic data verification was performed). In addition, the homolog compositions of the initial extraction and reextraction of the samples were compared and are presented in Figure 1. Since the original extraction and reextraction were performed using separate 1L sample volumes, the results for the repeat extraction and analyses were compared to the RAMP field duplicate criteria (The RPD for water field duplicates should be ≤35% for results >5x the RL. The difference between results should be ≤ the RL when at least one result is ≤5x the RL.)

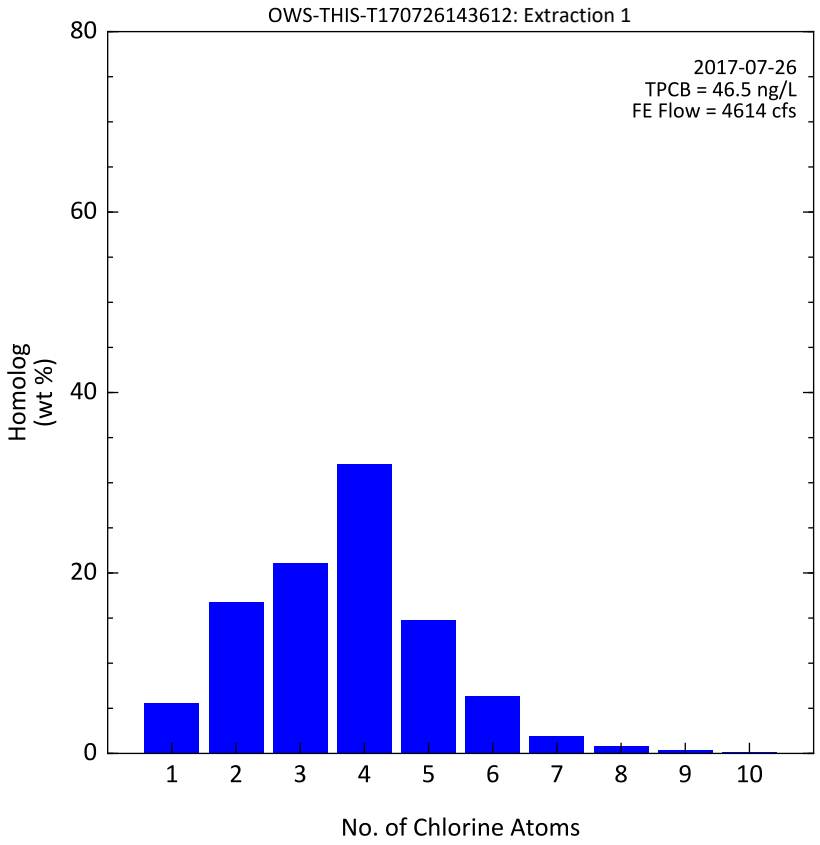
Table 2. Original Extraction/Analysis and Reextraction/Reanalysis Results

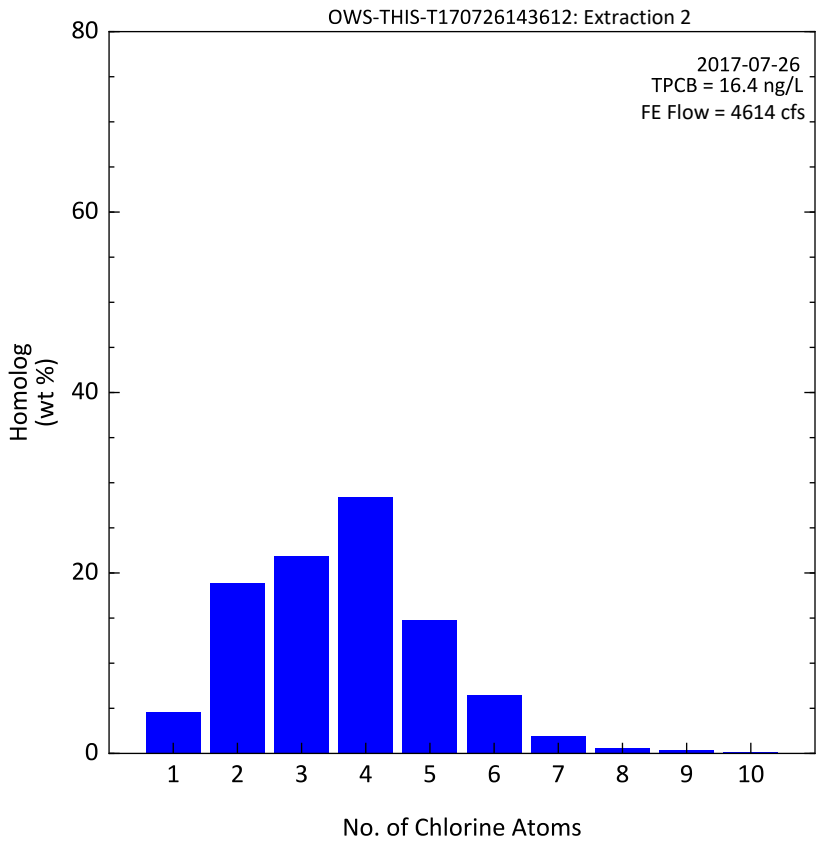
	Original Extraction/Analysis	Reextraction/ Reanalysis		Field Duplicate Criteria Met?
Sample ID	Total PCBs (ng/L)	Total PCBs (ng/L)	RPD	
OWS-THIS-T170726143612	46.5	16	98%	No

CONCLUSIONS

The results of the original extraction of sample OWS-THIS-T160823145340 were lower than the reextraction/reanalysis results; however, the original results were still inconsistent with the PCB results observed at TID during a similar timeframe. A comparison of the original extraction/analysis and reextraction/reanalysis results for sample OWS-THIS-T160823145340 revealed that the results were outside of the normal variability expected from two separate 1L sample volumes (98% RPD). However, similar homolog compositions were observed for the original extraction/analysis and reextraction/reanalysis (Figure 1).

The results of the original sample OWS-THIS-T160823145340 results were not confirmed by the reextraction and reanalysis. However, the homolog compositions of the initial and reextractions were generally consistent with one another. In addition, the elevated TSS result and the PCB homolog composition was consistent with the possibility of a resuspension event. Since both analyses met QC criteria (the extraction was repeated by Vista due to minor method blank contamination), both sets of results have been loaded into the RAMP database and now are identified as a field duplicate pair (the results of the original extraction are reported with the sample identification of OWS-BDUP-T170726143615).





Data source: \\nereus\e_drive\Projects\GE_Hudson\Dredging_Analysis\Working\Analysis\2018\Water\Homolog_Plots\Sample_OWS-THIS-T170726143612_2_Extractions.csv.
Notes: Duplicates are averaged. Non-detect values are excluded.

Publish Date: 04/04/2018 17:00 PM | User: WCL-MMAT
File Path: \\NEREUS\e_drive\Projects\GE_Hudson\Dredging_Analysis\Working\Analysis\2018\Water\Python\homolog_bySample.py

ANCHOR QEA

Figure 1
Vista Data - Homolog Patterns in Thompson Island

ENVIRONMENTAL STANDARDS
Setting the Standards for Innovative Environmental Solutions

HUDSON RIVER PCBs SITE

PHASE 2 REMEDIAL ACTION MONITORING PROGRAM

LABORATORY EVALUATION OF

PACE ANALYTICAL SERVICES, LLC.

GREEN BAY, WISCONSIN

August 23, 2019

Prepared for:

GENERAL ELECTRIC COMPANY
1 River Road – Bldg. 5-7W
Schenectady, NY 12345

Prepared by:

ENVIRONMENTAL STANDARDS, INC.
1140 Valley Forge Road
P.O. Box 810
Valley Forge, PA 19482-0810

On-site audits/evaluations of Pace Analytical Services, LLC. (Pace) located in Green Bay, Wisconsin, were conducted on February 13-14, 2018, by Mr. David R. Blye, CEAC and Ms. Meg A. Michell, M.S., and on April 12-13, 2018, by Mr. David R. Blye, CEAC of Environmental Standards, Inc. (hereafter referred to as the "Auditor"). These audits were performed on behalf of General Electric Company (GE) as part of the quality assurance efforts for the Hudson River PCBs Superfund Site Phase 2 Remedial Action Monitoring Program. The audits were performed in compliance with the Hudson River PCBs Superfund Site Phase 2 Remedial Action Monitoring Quality Assurance Project Plan (Phase 2 RAM QAPP, May 2012), Section 11.2.3. Specifically, the audit was performed for the following purposes:

- To assess the Pace facility with regard to capabilities and capacity relative to the analytical service needs of the Phase 2 RAMP.

- To provide Pace feedback on laboratory operating issues relative to compliance with the Phase 2 RAM QAPP, method compliance, SOP compliance, quality systems, and good laboratory practices.

- To evaluate Pace's understanding of, and conformance to, the technical and deliverable requirements stipulated in the Phase 2 RAM QAPP.

Facility Overview

Facility Name	Pace Analytical Services, LLC.
Address	1241 Bellevue Street, Suite 9 Green Bay, WI 54302
Phone Number	(920) 469-2436
Year facility founded	1992
Years in current location	21
Facility size	27,000 Square Feet
Number of personnel	~90 full time
Business hours	8:00am to 6:00pm CST M-F, 8:00am to 12:00pm Sat
National Environmental Laboratory Accreditation Program (NELAP) accrediting authority	Florida Department of Health
Date of last NELAP assessment	4/13/2016-4/15/2016
Certifications & accreditations	New York State Department of Health; NY Lab ID No. 12064
Laboratory Information Management System (LIMS)	Epic Pro (Horizon)

During the audit at Pace, interviews were conducted with the analysts and section managers to gain insight of the laboratory personnel's understanding of the analytical methods, standard operating procedures (SOPs), and the Phase 2 RAM QAPP technical and reporting

requirements. The Auditor evaluated the preparation and analysis of 2017 fish samples for Aroclor PCBs and lipids. The fish PCB data were generated by the Standard Operating Procedures (SOPs) for preparation and homogenization of tissue samples (SOP S-GB-L-009-Rev.00), extraction by SW-846 Method 3541 and determination of percent lipids (SOP S-GB-O-068-rev.00), and the analysis for PCB Aroclors using SW-846 Method 8082A (SOP S-GB-O-067-REV.00) included as Attachments 1, 2, and 3, respectively, of 2017 Corrective Action Memorandum No. 15 (CAM 15 August 15, 2017).

In addition, the following non-analytical areas of the laboratory facility were evaluated:

- Sample receipt and storage, including archival.
- Bottleware.
- Waste handling and storage.
- Quality documents including SOPs relevant to the scope of the audit.
- Record-keeping, including notebooks, logbooks, and logsheets.
- Data reporting.
- Data storage and archival, including hardcopy and electronic versions.
- Project management including documentation of client-specific requirements and communication and resolution of issues identified.
- Calibration records of testing equipment including fume hoods, weights, balances, National Institute of Science and Technology (NIST)-calibrated thermometer, and all temperature devices utilized in the facility.
- Quality Assurance (QA) systems including QC control limits and control charting; MDL studies and MDL determinations; QA training and training records; QA of electronic and hardcopy data; internal and external audits and corrective action tracking and implementation; and performance test results.

A tabular summary of the findings from the on-site audit is presented below. Each finding is numbered, categorized, ranked, and attributed to the specific analytical or non-analytical area(s) of the laboratory where the finding was observed. Findings are ranked as "**Critical**," "**Moderate**," or "**Minor**" according to their potential impact on data defensibility, data quality and usability, and laboratory or project operations. Critical findings require immediate corrective action by laboratory QA personnel. In addition, the tabular summary indicates whether the finding represents nonconformance with the Phase 2 RAM QAPP.

By the very nature of an audit, some critical statements that should not be regarded as an overall indication of the laboratory's qualifications are presented in this report; rather, these statements represent areas for laboratory operational and quality assurance (QA) improvement.

The laboratory audits identified six minor deficiencies and one critical deficiency in the various operational areas evaluated. The identified deficiencies can be corrected by Pace's management and technical personnel in conjunction with RAMP quality assurance oversight personnel through an overall review and improvement of the laboratory's Quality Assurance (QA) Program and current operational practices.

Findings Summary

#	Level	Applicable Area	Finding	Additional Detail
1	Minor	Fish Preparation	Larger fish that do not fit within the confines of the aluminum foil placed on the top loading balance need to be placed in a clean tray first so the fish does not hang over the edges of the top loading balance surface. The tray must be tarred on the balance prior to the fish being weighed.	
2	Minor	Fish Preparation	Modifications to the fish preparation Biota Homogenization Log were discussed to capture additional information to track grinder equipment used (chop, grind, Robo), number of containers of homogenate, and photo of filet on scale taken.	Subsequent to the onsite audit, Pace GB and Environmental Standards worked on created and electronic log that would capture the fish preparation information rather than recording the information on the Biota Homogenization Log. This electronic log would allow real time capture of the data and facilitate getting the select information into the GE fish database. The electronic record form was put into effect in 2019.
3	Minor	Extraction; Lipids	The balance used to weigh fish sample weights and lipids determinations was physically very far away from the extraction room. A balance should be placed within the extraction room to increase efficiency and to minimize potential impacts to contamination of lipids samples by addition of dust or particles to the aluminum weighing pans during moving the sample trays.	Until such time that a balance can be placed in the extraction room, the Auditors requested the lipids trays be covered with aluminum foil when mobilizing between rooms. Pace GB installed a balance in the extraction room in 2019.

#	Level	Applicable Area	Finding	Additional Detail
4	Minor	Extraction; Lipids	A syringe is used to obtain a 1mL portion of extract for the lipids analysis prior to cleanup of the extract for PCB analysis. The syringe is rinsed six (6) times with hexane contained in a 40 mL volatile vial between sample aliquots. The Auditors suggested the final rinse be in a separate vial of clean hexane instead of just the initial vial of hexane to minimize the final hexane rinse from becoming contaminated.	This request was implemented by Pace GB during the audit.
5	Minor	Extraction	The Auditors noted that when Aroclor and surrogate spikes were performed a spike witness was not present to observe per S-GB-O-68-Rev.00 Section 13.1.	This request was implemented by Pace GB during the audit.
6	Critical	Extraction	During the audit the initial batch of fish samples that were extracted appeared to have phase separation of the extract into two layers. This was reviewed and discussed during and after the audit. The phase layer was ultimately determined to be water.	After some experimentation, an additional sodium sulfate drying step was found to remove the water prior to final solvent exchange, reduction and concentration. All fish that were extracted prior to addition of this second drying step were reextracted for PCBs and Lipids analysis.
7	Comment	Lipids	The laboratory was requested to run the LCS sample for lipids to provide a second blank analysis since only Aroclor 1242 is spiked in the LCS. Additionally, the laboratory was requested to run the MS sample for lipids to provide a second duplicate analysis since only Aroclor 1242 is spiked in the MS.	This request was implemented by Pace GB during the audit, but this extra data could only be provided in the raw data package.
8	Minor	Fish Preparation	The Auditor observed during the preparation of 2017 Fall fish that the filet analyst was not always changing gloves between fish samples.	The filet analyst immediately complied with the request.

Appendix B
External Laboratory and Field Audits

(Provided on accompanying DVD)

ENVIRONMENTAL STANDARDS
Setting the Standards for Innovative Environmental Solutions

HUDSON RIVER PCBs SITE

PHASE 2 REMEDIAL ACTION MONITORING PROGRAM

LABORATORY EVALUATION OF

VISTA ANALYTICAL LABORATORY

EL DORADO HILLS, CALIFORNIA

August 23, 2019

Prepared for:

GENERAL ELECTRIC COMPANY
1 River Road – Bldg. 5-7W
Schenectady, NY 12345

Prepared by:

ENVIRONMENTAL STANDARDS, INC.
1140 Valley Forge Road
P.O. Box 810
Valley Forge, PA 19482-0810

An on-site audit/evaluation of Vista Analytical Laboratory (Vista) located in El Dorado Hills, California, was conducted on November 14, 2017, by Mr. David R. Blye, CEAC of Environmental Standards, Inc. (hereafter referred to as the "Auditor"). The audit was performed on behalf of General Electric Company (GE) as part of the quality assurance efforts for the Hudson River PCBs Superfund Site Phase 2 Remedial Action Monitoring Program. The audit was performed in compliance with the Hudson River PCBs Superfund Site Phase 2 Remedial Action Monitoring Quality Assurance Project Plan (Phase 2 RAM QAPP, May 2012), Section 11.2.3. Specifically, the audit was performed for the following purposes:

- To assess the Vista facility with regard to capabilities and capacity relative to the analytical service needs of the Phase 2 RAMP.

- To provide Vista feedback on laboratory operating issues relative to compliance with the Phase 2 RAM QAPP, method compliance, SOP compliance, quality systems, and good laboratory practices.

- To evaluate Vistas understanding of, and conformance to, the technical and deliverable requirements stipulated in the Phase 2 RAM QAPP.

Facility Overview

Facility Name	Vista Analytical Laboratory
Address	1104 Windfield Way El Dorado Hills, CA 95762
Phone Number	(916) 673-1520
Year facility founded	1990
Years in current location	29
Facility size	9,000 Square Feet
Number of personnel	~15 full time
Business hours	8-5 pm 5 days per week
National Environmental Laboratory Accreditation Program (NELAP) accrediting authority	Oregon
Date of last NELAP assessment	11/12/2015
Certifications & accreditations	New York State Department of Health; NY Lab ID No. 11411
Laboratory Information Management System (LIMS)	Element LIMs®

During the audit at Vista, interviews were conducted with the analysts and section managers to gain insight of the laboratory personnel's understanding of the analytical methods, standard operating procedures (SOPs), and the Phase 2 RAM QAPP technical and reporting requirements. The Auditor evaluated the preparation and analysis of aqueous samples for PCB Congeners. The PCB Congener data were generated by the Standard Operating Procedure

(SOP) for preparation and analysis of polychlorinated biphenyls (PCBs) by Method 1668A/C (SOP 31 Rev 15), included as Attachment 4 of 2017 Corrective Action Memorandum No. 14 (CAM 14 May 17, 2017).

In addition, the following non-analytical areas of the laboratory facility were evaluated:

- Sample receipt and storage, including archival.
- Bottleware.
- Waste handling and storage.
- Quality documents including SOPs relevant to the scope of the audit.
- Record-keeping, including notebooks, logbooks, and logsheets.
- Data reporting.
- Data storage and archival, including hardcopy and electronic versions.
- Project management including documentation of client-specific requirements and communication and resolution of issues identified.
- Calibration records of testing equipment including fume hoods, weights, balances, National Institute of Science and Technology (NIST)-calibrated thermometer, and all temperature devices utilized in the facility.
- Quality Assurance (QA) systems including QC control limits and control charting; MDL studies and MDL determinations; QA training and training records; QA of electronic and hardcopy data; internal and external audits and corrective action tracking and implementation; and performance test results.

A tabular summary of the findings from the on-site audit is presented below. Each finding is numbered, categorized, ranked, and attributed to the specific analytical or non-analytical area(s) of the laboratory where the finding was observed. Findings are ranked as "**Critical**," "**Moderate**," or "**Minor**" according to their potential impact on data defensibility, data quality and usability, and laboratory or project operations. Critical findings require immediate corrective action by laboratory QA personnel. In addition, the tabular summary indicates whether the finding represents nonconformance with the Phase 2 RAM QAPP.

By the very nature of an audit, some critical statements that should not be regarded as an overall indication of the laboratory's qualifications are presented in this report; rather, these statements represent areas for laboratory operational and quality assurance (QA) improvement.

The laboratory audit identified one minor deficiencies in the various operational areas evaluated. The identified deficiencies can be corrected by Vistas management and technical personnel in conjunction with RAMP quality assurance oversight personnel through an overall review and improvement of the laboratory's Quality Assurance (QA) Program and current operational practices.

Findings Summary

#	Level	Applicable Area	Finding	Additional Detail
1	Minor	Bottleware	The Auditor determined that the one liter (1L) amber glass sample containers supplied to the field for sample collection are not independently proofed by analysis to verify the containers are clean for trace level PCB congener analysis.	The 1L amber glass containers are purchased from Environmental Sampling Supply (ESS) as "pre-cleaned certified." Certification of cleanliness is provided with each case of containers. Additionally, Vista uses the same ESS glassware for preparation of the method blank for PCB congener analysis. This does provide a verification check of the sample container cleanliness. The auditor determined that an independent analysis was still preferred separately from the method blank that uses the same glassware, but that it was not mandatory for Vista to do so.

FIELD AUDIT REPORT

FISH MONITORING PROGRAM ACTIVITIES

PERFORMED ON AUGUST 28, 2017

HUDSON RIVER PCBs SITE

2017 REMEDIAL ACTION MONITORING PROGRAM

September 29, 2017

Prepared for:

GENERAL ELECTRIC COMPANY
381 Broadway
Building 40-2
Ft. Edward, NY 12828

Prepared by:

ENVIRONMENTAL STANDARDS, INC.
1140 Valley Forge Road
P.O. Box 810
Valley Forge, PA 19482-0810

Executive Summary

On August 28, 2017, Environmental Standards, Inc. (Environmental Standards) conducted an audit of field activities associated with fish sample collection and processing performed by Anchor QEA, LLC (Anchor QEA), and ARCADIS US, Inc. (Arcadis), personnel. The field audit performed by Environmental Standards identified that the members of the Anchor QEA and Arcadis Field Team conducted the work in a professional manner and complied with the majority of the procedures described in the Phase 2 Remedial Action Monitoring Quality Assurance Project Plan and associated standard operating procedures.

One audit finding was identified and is summarized below:

- Sample Packaging and Shipping
    - Temperature blanks were not present in the sample coolers during fish processing in the field. Temperature blanks should be placed into the sample coolers at the time the first investigative samples are placed in the coolers.

One finding identified during the August 2016 field audit had not yet been addressed; two of the weights used for daily scale calibration checks were expired and required re-certification. The Auditor suggested to the Field Team that the expired weights be submitted for third-party re-certification.

The Anchor QEA/Arcadis Field Team executed the field procedures effectively and expressed a positive attitude about quality assurance considerations for the project. Based on the Auditor's observations, the overall quality of services performed by the Field Team was satisfactory.

TABLE OF CONTENTS

1.0 Introduction

The purpose and background for the remediation of the Hudson River Polychlorinated Biphenyls (PCBs) Site are presented in the Record of Decision (ROD) issued by the United States Environmental Protection Agency (US EPA) in February 2002. The ROD provided for the dredging of specified sediments in the Upper Hudson River that meet certain criteria, as well as the establishment of certain performance standards to govern implementation of the remedial action. The ROD stipulated the performance of this remedial action in two phases. The first phase (Phase 1) was the first season of dredging operations, during which, dredging occurred at a reduced rate and extensive monitoring was conducted. Phase 1 was conducted in the Spring, Summer, and Fall of 2009. The second phase of the remedial action (Phase 2), which began in 2011, consisted of the remainder of the necessary dredging. Dredging was completed in 2015.

The Phase 2 Remedial Action Monitoring Program (RAMP) for 2017 generally includes the following monitoring components:

- Collection and analysis of water and fish samples for the purpose of determining the PCB concentrations that exist during remediation in the region of the Hudson River subject to remediation and in the broader region expected to benefit from the selected remedy.
- Monitoring of the suspended solids levels in water as an indicator of project-related sediment releases and monitoring of the levels of dissolved oxygen (DO) and pH in water to ascertain whether remediation is causing changes that could be harmful to aquatic biota or exceed drinking water standards.
- Collection and analysis of sediment samples to assess post-dredging residual PCB concentrations in accordance with the Addendum to the Surface Sediment Sampling and Work Plan for 2016 (Anchor QEA October 2017).

RAMP activities conducted during the performance of Phase 2 of the remedial action are governed by the Hudson River PCBs Superfund Site Phase 2 Remedial Action Monitoring Quality Assurance Project Plan (Phase 2 RAM QAPP, May 2012). The Phase 2 RAM QAPP addresses collection, analysis, and data management activities of samples collected in 2017 as part of Phase 2 of the remedial action. A revised version of Attachment A to the QAPP is currently being reviewed by US EPA. Corrective action memos (CAMs) are issued, as needed, to modify or amend the Phase 2 RAM QAPP. A Long-Term Operation, Maintenance, and Monitoring Plan for Water, Fish, and Sediment Monitoring (OMM Plan) is currently being prepared for the project and, upon approval, will supersede the Phase 2 RAM QAPP.

The General Electric Company (GE) retained Environmental Standards, Inc. (Environmental Standards), to conduct field audits of specific field activities associated with the implementation of the Phase 2 RAMP. Specifically, the subject of this Field Audit Report is the field audit (performed by Environmental Standards) of activities associated with implementation of the Fish Monitoring Program. The audited activities were conducted by GE's environmental contractor, Anchor QEA, LLC (Anchor QEA), with support from ARCADIS US, Inc. (Arcadis).

This audit was performed to evaluate and document the overall quality of field services provided by the contractors (Anchor QEA and Arcadis) and to verify conformance of field procedures with project requirements as specified in the project control documents (*i.e.*, Phase 2 RAM QAPP, the associated standard operating procedures [SOPs], and CAMs).

On August 28, 2017, Environmental Standards representative Joseph P. Kraycik (the "Auditor") conducted an audit of field activities associated with fish sample collection and processing performed by Anchor QEA and Arcadis personnel. The objective of the audited activities was to collect representative fish samples from pre-determined locations in the Upper Hudson River at the Thompson Island and Fort Miller/Northumberland Pools. The audit by Environmental Standards focused on an evaluation of several important aspects of the field activities being performed on those days. The following general headings were used to group field-audit activities for evaluation purposes:

1. Pre-Task Planning, Coordination, and Management
2. Field Documentation and Records
3. Supplies, Equipment, and Preparation
4. Fish Sampling Procedures
5. Decontamination and Waste Management Procedures
6. Quality Assurance/Quality Control Sample Collection
7. Sample Packaging and Shipping
8. Sample Custody
9. Training and Health & Safety
10. General Observations

During the field audit, the Auditor utilized a field audit checklist that was developed specifically for the Fish Monitoring Program field activities. This checklist contained pertinent items under each of the previously cited headings and provided the basic guidelines for a comprehensive audit.

Due to the nature of a field audit, some critical statements are presented in this report. These statements are based on observations made in the field and address those areas in which project Field Team deficiencies were noted and/or where changes may be appropriate. An exhaustive list of those activities performed in accordance with the project control documents and observed to be in compliance with standard industry protocol is not presented in this audit report; rather, this report identifies the type and source of deviations from the primary project control document (Phase 2 RAM QAPP, which contains the relevant SOPs for the project) as well as industry standards/best management practices.

2.0 Field Activities

On August 28, 2017, the Auditor observed fish sampling activities conducted at five sampling sites within the Thompson Island Pool (representative of River Section 1) and two sampling sites within the Northumberland/Fort Miller Pool (representative of River Section 2) of the Upper Hudson River. Fish sampling activities were performed during the daylight hours and targeted yearly pumpkinseed and resident forage fish.

Fish sampling activities observed during the audit were a collective effort conducted by the Field Team. Anchor QEA personnel (Jim Ryan, Rich Constant, and Scott Andrews) were responsible for fish sample collection and processing duties that occurred onshore. Arcadis personnel (Dave Rigg and Jason Vogel) were responsible for operation of the electroshocking boat and fish sample collection duties. Regulatory oversight was performed during the sampling event by Tom Shinskey of the Louis Berger Group on behalf of US EPA. Oversight was not performed on behalf of New York State Department of Environmental Conservation (NYS DEC) during the field audit.

The goal of the fish sampling event was to collect five individual pumpkinseed specimens at the "TD-1 through TD-4" and at the "ND-1 and ND-2" locations and 10 individual specimens at the "TD-5" location using electroshocking apparatus. In addition, two composite samples consisting of multiple forage fish were collected at each location. Forage fish consisted of spottail, spotfin, golden, and bluntnose shiner. The target number of specimens for each species was achieved at each location with the exception of ND-2 where only four pumpkinseed samples were collected. In order to account for this shortage, a sixth pumpkinseed specimen was collected at the ND-1 location.

The Auditor accompanied the sampling crew on the vessel during electrofishing at the TD collection areas. The electroshocking vessel was operated along transects and shoreline/cove areas and the stunned fish were recovered using dip nets. Fish meeting species and size criteria established in the Phase 2 RAM QAPP were placed into a live-well (filled with river water) awaiting processing; fish specimens not meeting established size criteria were released back into the river. Fish specimens collected at the Thompson Island Pool were transported to the public boat launch near Fort Edward, New York, and transferred to Anchor QEA onshore personnel for processing. Fish specimens collected at the Northumberland/Fort Miller Pool were transported to the Anchor QEA field office in Saratoga Springs, New York, for processing.

Fish specimen processing consisted of collection and documentation of data (including species identification, size, weight, and observed abnormalities, as well as water quality data), collection of scale and spine samples in sample envelopes, packaging of fish body samples in aluminum foil and plastic resealable bags, labeling of fish scale and fish body samples, and placement of fish specimen samples into coolers with bagged wet ice for temperature preservation. Data obtained during fish specimen processing were entered into individual electronic field data logs that were uploaded into the project database. Fish specimen sample labels, Chain-of-Custody (COC) forms, and Field Logs were generated using the database records.

Fish specimen samples were packaged for pick-up and delivery to the Pace Analytical Services, Inc. (Pace), laboratory in Schenectady, New York, for analysis of PCBs (by US EPA SW-846 Method 8082 Aroclor Sum Method) and percent lipids. Mr. Ryan stated that a Pace courier would pick up the samples the following day at the Anchor QEA Saratoga Springs field office. The Auditor did not observe final sampling packaging or sealing of the coolers prior to shipment.

Upon receipt in Schenectady, the samples were then to be frozen at the Pace service center and later shipped to the Pace Green Bay laboratory for analysis of PCBs (by US EPA SW-846 Method 8082 Aroclor Sum Method) and percent lipids.

Field activities performed by the Anchor QEA and Arcadis Field Team and observed by the Auditor during the performance of fish sampling activities were consistent with those previously described, except as otherwise noted.

3.0 Audit Findings

During the field activities, the Auditor observed the Field Team collect and process fish samples to evaluate field performance and compliance with the project control documents. Observations of field procedures were recorded on a field audit checklist. The following sections provide the results of the audit and follow the major headings presented in Section 1.

3.1 Pre-Task Planning, Coordination, and Management

The Auditor arrived to the project site shortly after field activities had begun on the morning of August 28, 2017. Field Team members briefed the Auditor on activity-specific topics and health and safety protocol upon the Auditor's arrival.

The Auditor verified that the Field Team was familiar with the project control documents, objectives, and scope prior to implementation of field activities. Copies of the project control documents were available for reference during field activities. The Field Team was observed to be generally well prepared for the field activities and had properly planned and coordinated activities with appropriate project personnel.

No deficiencies associated with pre-task planning, coordination, and management were observed or reported.

3.2 Field Documentation and Records

Field documentation maintained by the Field Team (for sample collection and processing tasks) primarily consisted of individual electronic field data logs that were completed on a field laptop computer and uploaded into the electronic project database. The electronic logs were used to record specific field measurements, data, and pertinent observations associated with the fish samples. Equipment calibrations/checks were documented on project-specific forms (provided to the Auditor for review) and in field logbook (maintained by Mr. Ryan). Individual sample labels, collective sample COC forms, and field log forms were generated using the field database.

No deficiencies associated with field documentation and records were observed or reported.

3.3 Supplies, Equipment, and Preparation

The Auditor verified that the field equipment used during fish sample collection and processing activities was consistent with the requirements of the Phase 2 RAM QAPP. The Field Team reported to the Auditor that field equipment was inspected daily and maintenance was performed on an as-needed basis by trained personnel in accordance with the Phase 2 RAM QAPP. The Auditor observed that a sufficient supply of sampling and processing equipment and materials were available for use during the field activities.

The YSI 6920 Multi-Parameter Water Quality Sonde was calibrated prior to use by the rental company (Pine Environmental) that supplied the instrument and was documented on an equipment checklist form that was reviewed by the Auditor. The My Weigh i500 electronic balance was checked prior to use by weighing four certified weights (*i.e.*, 2, 10, 50, and 100 grams) and the results were documented by the Field Team. The balance checks were documented on a Weight Check Calibration Log that was reviewed by the Auditor.

3.4 Fish Sampling Procedures

The Auditor observed the sampling procedures performed by the Field Team during the fish sample collection event on August 29, 2016. The collection and processing of fish specimen samples (as discussed in the Field Activities section of this report) were performed in a consistent manner during the field audit. With the exception of the fifth pumpkinseed specimen at location ND-2, all the target number of fish samples were readily obtained and collected in a timely fashion.

No deficiencies were observed or reported.

3.5 Decontamination and Waste Management Procedures

Disposable materials (*e.g.,* paper towels, nitrile gloves) used during collection and specimen processing activities were bagged for proper disposal. The measuring table and plastic container used to hold specimens during weighing were wiped down with a paper towel after each specimen was measured and weighed. Cleaning of the measuring table and plastic container used to weigh the specimens consisted of wiping down each item with a paper towel after each use. A more thorough decontamination was conducted by rinsing the items with river water after each sampling location and at the conclusion of the day's activities. Disposable materials used during sampling activities were bagged for proper disposal.

No deficiencies associated with decontamination and waste management procedures were observed or reported.

3.6 Quality Assurance/Quality Control Sample Collection

The Auditor observed collection of quality assurance/quality control (QA/QC) samples during the field sampling/processing activities, including matrix spike (MS) and laboratory duplicate (LD) samples. The Field Team reported to the Auditor that QA/QC sample collection activities were performed in compliance with procedural and frequency requirements specified in the Phase 2 RAM QAPP. QA/QC sample collection activities observed by the Auditor appeared to be consistent with the requirements of the Phase 2 RAM QAPP. On August 28, 2017, one forage fish composite sample from TD-1 and two forage fish composite samples from ND-2 were designated on the COC forms as MS/LD samples.

No deficiencies associated with QA/QC sample collection were observed or reported.

3.7 Sample Packaging and Shipping

The Auditor observed sample packaging activities conducted by the Field Team during the sample processing portion of the audit. Once sample processing activities were completed, the whole fish (pumpkinseed) and composite (forage fish) samples were packaged into laboratory coolers with sufficient bagged wet ice. Mr. Ryan stated that the Field Team was going to add ice and repackage the coolers as necessary prior to pick up of the cooler by the Pace courier.

The Auditor did not observe final sealing of the coolers or the use of custody seals for transport to the analytical laboratory on August 29, 2017; however, based on information reported by the Field Team, the required procedures, as presented in the Phase 2 RAM QAPP, were being followed.

Finding: Temperature blanks were not present in the coolers during fish processing in the field. It was reported to the Auditor that temperature blanks would be added to the coolers later in the afternoon upon return to the Anchor QEA field office.

Recommendation: Temperature blanks should be placed into the sample coolers at the time the first investigative samples are placed in the coolers.

3.8 Sample Custody

The Auditor observed the Field Team maintain proper sample custody from the time of collection through processing and packaging; although, final sample relinquishment to the laboratory was not observed by the Auditor. In addition, the Auditor reviewed copies of COC documentation from the day's sampling activities, which appeared to have been completed in accordance with Phase 2 RAM QAPP requirements. An MS/LD sample was assigned to each COC.

No deficiencies associated with sample custody were observed or reported.

3.9 Training and Health & Safety

The Field Team reported to the Auditor that each Field Team member had successfully completed the required project health and safety training and project task-specific training as necessary and that each Field Team member possessed current Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) training certifications under 29 CFR 1910.120.

The Auditor verified that the Field Team was familiar with the project health and safety plan (HASP) and that a copy of the HASP was available for reference during field activities. The Auditor observed that general health and safety procedures were followed during the field audit and that proper personal protective equipment (PPE) was available and in use by the Field Team.

No deficiencies were observed or reported.

3.10 General Observations

The Field Team appeared to be well trained and was familiar with the protocols for sample collection and processing. Field Team personnel were well organized and conducted field activities in a professional manner.

3.11 2016 Field Audit Findings

One finding was initially identified during the August 29, 2016 field audit and has not yet been addressed:

Finding: The certification for the 10- and 100-gram weights used for scale calibration checks had expired.

Recommendation: The 10- and 100-gram weights should be submitted to a qualified third-party vendor for re-certification.

During the June 2017 field audit, the Field Team reported that the 10- and 100-gram weights have not been re-certified. The Auditor recommended that the weights be submitted for re-certification.

4.0 Conclusions

The field audit performed by Environmental Standards identified that the Field Team conducted its work in a professional manner and complied with the procedures described in the Phase 2 RAM QAPP and associated SOPs.

One audit finding was identified and is summarized below:

- Sample Packaging and Shipping
    - Temperature blanks were not present in the sample coolers during fish processing in the field. Temperature blanks should be placed into the sample coolers at the time the first investigative samples are placed in the coolers.

The finding and recommendation identified in this audit report were discussed with the Field Team at the conclusion of the audit. The deficiency is generally minor in nature, easily corrected, would not likely have resulted in a major impact to sample quality, and would not likely have jeopardized the data quality objectives of the project. The deficiency discussed herein has the potential to compromise sample integrity and, therefore, should be rectified as recommended in this report.

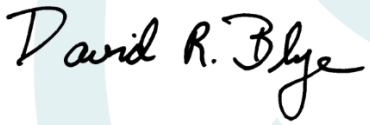
One finding identified during the August 2016 field audit had not yet been addressed; the 10- and 100-gram weights used for daily scale calibration checks were expired and required re-certification. The Auditor suggested to the Field Team that the expired weights be submitted for third-party re-certification.

The Field Team executed the field procedures effectively and expressed a positive attitude about QA considerations for the project. Based on the Auditor's observations, the overall quality of services performed by the Field Team was satisfactory.

Audit Performed by,

Joseph P. Kraycik, P.G., CQA
Consulting Geoscientist

Report Reviewed by,



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Date: September 29, 2017

FIELD AUDIT REPORT

FISH MONITORING PROGRAM ACTIVITIES

PERFORMED ON JUNE 14, 2017

HUDSON RIVER PCBs SITE

2017 REMEDIAL ACTION MONITORING PROGRAM

July 24, 2017

Prepared for:

GENERAL ELECTRIC COMPANY
381 Broadway
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Prepared by:

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Executive Summary

On June 14, 2017, Environmental Standards, Inc. (Environmental Standards) conducted an audit of field activities associated with fish sample collection and processing performed by Anchor QEA, LLC (Anchor QEA), and ARCADIS US, Inc. (ARCADIS), personnel. The field audit performed by Environmental Standards identified that the members of the Anchor QEA and ARCADIS Field Team conducted the work in a professional manner and complied with the majority of the procedures described in the Phase 2 Remedial Action Monitoring Quality Assurance Project Plan and associated standard operating procedures.

One finding identified during the August 2016 field audit had not yet been addressed; two of the weights used for daily scale calibration checks were expired and required re-certification. The Auditor suggested to the Field Team that the expired weights be submitted for third-party re-certification.

The Anchor QEA/ARCADIS Field Team executed the field procedures effectively and expressed a positive attitude about quality assurance considerations for the project. Based on the Auditor's observations, the overall quality of services performed by the Field Team was satisfactory.

TABLE OF CONTENTS

1.0 Introduction

The purpose and background for the remediation of the Hudson River Polychlorinated Biphenyls (PCBs) Site are presented in the Record of Decision (ROD) issued by the United States Environmental Protection Agency (US EPA) in February 2002. The ROD provided for the dredging of specified sediments in the Upper Hudson River that meet certain criteria, as well as the establishment of certain performance standards to govern implementation of the remedial action. The ROD stipulated the performance of this remedial action in two phases. The first phase (Phase 1) was the first season of dredging operations, during which, dredging occurred at a reduced rate and extensive monitoring was conducted. Phase 1 was conducted in the Spring, Summer, and Fall of 2009. The second phase of the remedial action (Phase 2), which began in 2011, consists of the remainder of the necessary dredging. Although dredging was completed in 2015, habitat restoration work continues and is considered remedial action.

The Phase 2 Remedial Action Monitoring Program (RAMP) for 2017 generally includes the following monitoring components:

- Collection and analysis of water and fish samples for the purpose of determining the PCB concentrations that exist during remediation in the region of the Hudson River subject to remediation and in the broader region expected to benefit from the selected remedy.
- Monitoring of the suspended solids levels in water as an indicator of project-related sediment releases and monitoring of the levels of dissolved oxygen (DO) and pH in water to ascertain whether remediation is causing changes that could be harmful to aquatic biota or exceed drinking water standards.
- Collection and analysis of sediment samples to assess post-dredging residual PCB concentrations in accordance with the Phase 2 Residuals Performance Standard and the 2011 Performance Standards Compliance Plan (PSCP).

RAMP activities conducted during the performance of Phase 2 of the remedial action are governed by the Hudson River PCBs Superfund Site Phase 2 Remedial Action Monitoring Quality Assurance Project Plan (Phase 2 RAM QAPP, May 2012). The Phase 2 RAM QAPP addresses collection, analysis, and data management activities of samples collected in 2017 as part of Phase 2 of the remedial action. A revised version of Attachment A to the QAPP is currently being reviewed by US EPA. Corrective action memos (CAMs) are issued, as needed, to modify or amend the Phase 2 RAM QAPP. A Long-Term Operation, Maintenance, and Monitoring Plan for Water, Fish, and Sediment Monitoring (OMM Plan) is currently being prepared for the project and, upon approval, will supersede the Phase 2 RAM QAPP.

The General Electric Company (GE) retained Environmental Standards, Inc. (Environmental Standards), to conduct field audits of specific field activities associated with the implementation of the Phase 2 RAMP. Specifically, the subject of this Field Audit Report is the field audit (performed by Environmental Standards) of activities associated with implementation of the Fish Monitoring Program. The audited activities were conducted by GE's environmental contractor, Anchor QEA, LLC (Anchor QEA), with support from ARCADIS US, Inc. (ARCADIS).

This audit was performed to evaluate and document the overall quality of field services provided by the contractors (Anchor QEA and ARCADIS) and to verify conformance of field procedures with project requirements as specified in the project control documents (*i.e.*, Phase 2 RAM QAPP, the associated standard operating procedures [SOPs], and CAMs).

On June 14, 2017, Environmental Standards representative Joseph P. Kraycik (the "Auditor") conducted an audit of field activities associated with fish sample collection and processing performed by Anchor QEA and ARCADIS personnel. The objective of the audited activities was to collect representative fish samples from pre-determined locations in the Upper Hudson River at the Stillwater Pool. The audit by Environmental Standards focused on an evaluation of several important aspects of the field activities being performed on those days. The following general headings were used to group field-audit activities for evaluation purposes:

1. Pre-Task Planning, Coordination, and Management
2. Field Documentation and Records
3. Supplies, Equipment, and Preparation
4. Fish Sampling Procedures
5. Decontamination and Waste Management Procedures
6. Quality Assurance/Quality Control Sample Collection
7. Sample Packaging and Shipping
8. Sample Custody
9. Training and Health & Safety
10. General Observations

During the field audit, the Auditor utilized a field audit checklist that was developed specifically for the Fish Monitoring Program field activities. This checklist contained pertinent items under each of the previously cited headings and provided the basic guidelines for a comprehensive audit.

Due to the nature of a field audit, some critical statements are presented in this report. These statements are based on observations made in the field and address those areas in which project Field Team deficiencies were noted and/or where changes may be appropriate. An exhaustive list of those activities performed in accordance with the project control documents and observed to be in compliance with standard industry protocol is not presented in this audit report; rather, this report identifies the type and source of deviations from the primary project control document (Phase 2 RAM QAPP, which contains the relevant SOPs for the project) as well as industry standards/best management practices.

2.0 Field Activities

On June 14, 2017, the Auditor observed fish sampling activities conducted at the Stillwater Pool (representative of River Section 2) sampling locations in the Upper Hudson River. Fish sampling activities were performed during the daylight hours and targeted three fish species – black bass, perch, and bullhead.

Fish sampling activities observed during the audit were a collective effort conducted by the Field Team. Anchor QEA personnel (Jim Ryan, Rich Constant, and Matt Smith) were responsible for fish sample collection and processing duties that occurred on shore. ARCADIS personnel (Dave Rigg and Jason Vogel) were responsible for operation of the electroshocking boat and fish sample collection duties. Regulatory oversight was performed during the sampling event by Tom Shinskey of the Louis Berger Group on behalf of US EPA. Oversight was not performed on behalf of New York State Department of Environmental Conservation (NYS DEC) during the field audit.

The goal of the fish sampling event was to collect 10 individual specimens of each target species at the "SW3" location and five individual specimens of each target species at the "SW2," "SW4," and "SW5" locations using electroshocking apparatus. The target number of specimens for perch and bullhead was achieved at each collection location during the field audit.

The Auditor accompanied the sampling crew on the vessel during electrofishing at the SW2 collection area. The electroshocking vessel was operated along transects and shoreline/cove areas and the stunned fish were recovered using dip nets. Fish meeting species and size criteria established in the Phase 2 RAM QAPP were placed into a live-well (filled with river water) awaiting processing; fish specimens not meeting established size criteria were released back into the river. Collected fish specimens at the Stillwater Pool were transported to the parking lot at the Alcove Marina (for SW2 and SW3) and Stillwater Riverfront Park (for SW4 and SW5) and transferred to Anchor QEA onshore personnel for processing.

Fish specimen processing consisted of collection and documentation of data (including species identification, size, weight, and observed abnormalities, as well as water quality data), collection of scale and spine samples in sample envelopes, packaging of fish body samples in aluminum foil and plastic resealable bags, labeling of fish scale and fish body samples, and placement of fish specimen samples into coolers with bagged wet ice for temperature preservation. Data obtained during fish specimen processing were entered into individual electronic field data logs that were uploaded into the project database. Fish specimen sample labels, Chain-of-Custody (COC) forms, and Field Logs were generated using the database records.

Due to recent commercial carrier shipping issues, Mr. Ryan stated that samples collected during the field audit would be packaged for delivery to the Pace Analytical Services, LLC (Pace) service center in Schenectady, New York, on June 15, 2017. Samples would then be frozen at the Pace service center and later shipped to the Pace Green Bay laboratory for analysis of PCBs (by US EPA SW-846 Method 8082 Aroclor Sum Method) and percent lipids.

Field activities performed by the Anchor QEA and ARCADIS Field Team and observed by the Auditor during the performance of fish sampling activities were consistent with those previously described, except as otherwise noted.

3.0 Audit Findings

During the field activities, the Auditor observed the Field Team collect and process fish samples to evaluate field performance and compliance with the project control documents. Observations of field procedures were recorded on a field audit checklist. The following sections provide the results of the audit and follow the major headings presented in Section 1.

3.1 Pre-Task Planning, Coordination, and Management

The Auditor attended the daily pre-task meeting held by the Field Team that addressed topics such as the project scope and objectives, identification of personnel and responsibilities, and health and safety issues. The Field Team debriefed the Auditor on field activity-specific topics and health and safety protocol during pre-task meetings between the Field Team and the Auditor prior to the start of field activities.

The Auditor verified that the Field Team was familiar with the project control documents, objectives, and scope prior to implementation of field activities. Copies of the project control documents were available for reference during field activities. The Field Team was observed to be generally well prepared for the field activities and had properly planned and coordinated activities with appropriate project personnel.

No deficiencies associated with pre-task planning, coordination, and management were observed or reported.

3.2 Field Documentation and Records

Field documentation maintained by the Field Team (for sample collection and processing tasks) primarily consisted of individual electronic field data logs that were completed on a field laptop computer and uploaded into the electronic project database. The electronic logs were used to record specific field measurements, data, and pertinent observations associated with the fish samples. Equipment calibrations/checks were documented on project-specific forms and in field logbooks as described in Section 3.3. Individual sample labels, collective sample COC forms, and field log forms were generated using the field database.

A secondary form of field documentation utilized by some members of the Field Team included bound, dedicated project field logbooks. The logbooks were utilized to record brief details or notes pertaining to daily field activities. Although the Phase 2 RAM QAPP does not specifically require the use of a logbook for field documentation purposes, the use of such a record is beneficial to the project by providing a stand-alone record of pertinent data and details generated during field activities and also allows for the documentation of relevant information that cannot be stored in the project database under the current format. Use of a logbook for field documentation purposes is a standard industry practice and the Auditor encourages the Field Team to continue the use of a field logbook as part of the field documentation process.

No deficiencies associated with field documentation and records were observed or reported.

3.3 Supplies, Equipment, and Preparation

The Auditor verified that the field equipment used during fish sample collection and processing activities was consistent with the requirements of the Phase 2 RAM QAPP. The Field Team reported to the Auditor that field equipment was inspected daily and maintenance was

performed on an as-needed basis by trained personnel in accordance with the Phase 2 RAM QAPP. The Auditor observed that a sufficient supply of sampling and processing equipment and materials were available for use during the field activities.

The OHaus® Valor™ 2000 electronic balance (used to weigh bullhead, perch, and black bass) was checked prior to use by weighing a series of certified weights ranging from 100 grams to 2,000 grams. The balance checks were documented on a Weight Check Calibration Log that was reviewed by the Auditor. The YSI 6920 Multi-Parameter Water Quality Sonde was calibrated prior to use by the rental company (Pine Environmental) that supplied the instrument and was documented on an equipment calibration form that was reviewed by the Auditor.

As described in further detail in Section 3.11 of this Audit Report, two of the weights used for calibration checks were expired. This issue was identified during an August 2017 field audit and had not yet been addressed.

3.4 Fish Sampling Procedures

The Auditor observed the sampling procedures performed by the Field Team during the fish sample collection event on June 14, 2017. The collection and processing of fish specimen samples (as discussed in the Field Activities section of this report) were performed in a consistent manner during the field audit. Field sampling activities were generally conducted in accordance with project control document requirements and industry standards.

No deficiencies associated with field sampling procedures were observed or reported.

3.5 Decontamination and Waste Management Procedures

Disposable materials (*e.g.,* paper towels, nitrile gloves) used during collection and specimen processing activities were bagged for proper disposal. The measuring table and plastic container used to hold specimens during weighing were wiped down with a paper towel after each specimen was measured and weighed. Cleaning of the measuring table and plastic container used to weigh the specimens consisted of wiping down each item with a paper towel after each use. A more thorough decontamination was conducted by rinsing the items with river water after each sampling location and at the conclusion of the day's activities. Disposable materials used during sampling activities were bagged for proper disposal.

No deficiencies associated with decontamination and waste management procedures were observed or reported.

3.6 Quality Assurance/Quality Control Sample Collection

The Auditor observed collection of quality assurance/quality control (QA/QC) samples during the field sampling/processing activities, including matrix spike (MS) and laboratory duplicate (LD) samples. The Field Team reported to the Auditor that QA/QC sample collection activities were performed in compliance with procedural and frequency requirements specified in the Phase 2 RAM QAPP. QA/QC sample collection activities observed by the Auditor appeared to be consistent with the requirements of the Phase 2 RAM QAPP. On June 14, 2017, one black bass, two brown bullhead, and one yellow perch sample from the Stillwater Pool were designated on the COC forms as MS/LD samples.

No deficiencies associated with QA/QC sample collection were observed or reported.

3.7 Sample Packaging and Shipping

The Auditor observed sample packaging activities conducted by the Field Team during the sample processing portion of the audit. Once sample processing activities were completed, the whole fish samples were packaged into laboratory coolers with sufficient bagged wet ice and a temperature blank. Mr. Ryan stated that the Field Team was going to add ice and repackage the coolers as necessary prior to delivery of the sample coolers to the Pace service center.

The Auditor did not observe final sealing of the coolers or the use of custody seals for transport to the analytical laboratory on June 16, 2017; however, based on information reported by the Field Team, the required procedures, as presented in the Phase 2 RAM QAPP, were being followed.

No deficiencies associated with sample packaging or shipping were observed or reported.

3.8 Sample Custody

The Auditor observed the Field Team maintain proper sample custody from the time of collection through processing and packaging; although, final sample relinquishment to the laboratory was not observed by the Auditor. In addition, the Auditor reviewed copies of COC documentation from the day's sampling activities, which appeared to have been completed in accordance with Phase 2 RAM QAPP requirements. An MS/LD sample was assigned to each COC.

No deficiencies associated with sample custody were observed or reported.

3.9 Training and Health & Safety

The Field Team reported to the Auditor that each Field Team member had successfully completed the required project health and safety training and project task-specific training as necessary and that each Field Team member possessed current Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) training certifications under 29 CFR 1910.120.

The Auditor verified that the Field Team was familiar with the project health & safety plan (HASP) and that a copy of the HASP was available for reference during field activities. Health and safety issues were addressed during pre-task meetings held between the Auditor and Field Team (discussed in Section 3.1 of this report) the morning of the field audit. The Auditor observed that general health and safety procedures were followed during the field audit and that proper personal protective equipment (PPE) was available and in-use by the Field Team.

No deficiencies were observed or reported.

3.10 General Observations

The Field Team appeared to be well trained and was familiar with the protocols for sample collection and processing. Field Team personnel were well organized, conducted field activities in a professional manner, and were receptive to recommendations made by the Auditor.

3.11 2016 Field Audit Findings

One finding was identified during the August 29, 2016 field audit:

Finding: The certification for the 10- and 100-gram weights used for scale calibration checks had expired.

Recommendation: The 10- and 100-gram weights should be submitted to a qualified third-party vendor for re-certification.

During the June 2017 field audit, the Field Team reported that the 10 and 100-gram weights have not been re-certified. The Auditor recommended that the weights be submitted for re-certification.

4.0 Conclusions

The field audit performed by Environmental Standards identified that the Field Team conducted its work in a professional manner and complied with the procedures described in the Phase 2 RAM QAPP and associated SOPs.

One finding identified during the August 2016 field audit had not yet been addressed; two of the weights used for daily scale calibration checks were expired and required re-certification. The Auditor suggested to the Field Team that the expired weights be submitted for third-party re-certification.

The Field Team executed the field procedures effectively and expressed a positive attitude about quality assurance considerations for the project. Based on the Auditor's observations, the overall quality of services performed by the Field Team was satisfactory.

Audit Performed by,

Joseph P. Kraycik, P.G., CQA
Consulting Geoscientist

Report Reviewed by,

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Principal Chemist

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Date: July 24, 2017

FIELD AUDIT REPORT

OFF-SEASON WATER MONITORING

PERFORMED ON NOVEMBER 1, 2017

HUDSON RIVER PCBs SITE

2017 REMEDIAL ACTION MONITORING PROGRAM

November 28, 2017

Prepared for:

GENERAL ELECTRIC COMPANY
381 Broadway
Building 40-2
Ft. Edward, NY 12928

Prepared by:

ENVIRONMENTAL STANDARDS, INC.
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Executive Summary

The field audit performed by Environmental Standards, Inc., identified that the Anchor QEA, LLC, Field Team conducted its work in a professional manner and complied with the procedures described in the Phase 2 Remedial Action Monitoring Quality Assurance Project Plan and associated standard operating procedures.

The Field Team executed the field procedures effectively and expressed a positive attitude about QA considerations for the project. Based on the Auditor's observations, the overall quality of services performed by the Field Team was satisfactory.

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1.0 Introduction

The purpose and background for the remediation of the Hudson River Polychlorinated Biphenyls (PCBs) Site are presented in the Record of Decision (ROD) issued by the United States Environmental Protection Agency (US EPA) in February 2002. The ROD provided for the dredging of specified sediments in the Upper Hudson River that meet certain criteria, as well as the establishment of certain performance standards to govern implementation of the remedial action. The ROD stipulated the performance of this remedial action in two phases. The first phase (Phase 1) was the first season of dredging operations, during which, dredging occurred at a reduced rate and extensive monitoring was conducted. Phase 1 was conducted in the Spring, Summer, and Fall of 2009. The second phase of the remedial action (Phase 2), which began in 2011, consists of the remainder of the necessary dredging. Dredging was completed in 2015.

The Phase 2 Remedial Action Monitoring Program (RAMP) for 2017 generally includes the following monitoring components:

- Collection and analysis of water and fish samples for the purpose of determining the PCB concentrations that exist during remediation in the region of the Hudson River that is subject to remediation and in the broader region expected to benefit from the selected remedy.
- Monitoring of the suspended solids levels in water as an indicator of project-related sediment releases and monitoring of the levels of dissolved oxygen (DO) and pH in water to ascertain whether remediation is causing changes that could be harmful to aquatic biota or exceed drinking water standards.
- Collection and analysis of sediment samples to assess post-dredging residual PCB concentrations in accordance with the Addendum to the Surface Sediment Sampling and Work Plan for 2016 (Anchor QEA October 2017).
- .

RAMP activities conducted during the performance of Phase 2 of the remedial action are governed by the Hudson River PCBs Superfund Site Phase 2 Remedial Action Monitoring Quality Assurance Project Plan (Phase 2 RAM QAPP, May 2012). The Phase 2 RAM QAPP addresses collection, analysis, and data management activities of samples collected in 2017 as part of Phase 2 of the remedial action. A revised version of Attachment A to the RAM QAPP is currently being reviewed by US EPA. Corrective Action Memos (CAMs) are issued, as needed, to modify or amend the Phase 2 RAM QAPP. A Long-Term Operation, Maintenance, and Monitoring Plan for Water, Fish, and Sediment Monitoring (OMM Plan) is currently being prepared for the project and, upon approval, will supersede the Phase 2 RAM QAPP.

The General Electric Company (GE) retained Environmental Standards, Inc. (Environmental Standards), to conduct audits of specific field activities associated with the implementation of the Phase 2 RAMP. Specifically, the subject of this Field Audit Report is the audit (performed by Environmental Standards) of sampling and monitoring activities associated with the following program:

- Off-Season Water Column Monitoring

The audited activities were conducted by GE's environmental contractor, Anchor QEA, LLC (Anchor QEA).

This audit was performed to evaluate and document the overall quality of field services provided by the contractor (Anchor QEA) and to verify conformance of field procedures with project requirements as specified in the project control documents (*i.e*., Phase 2 RAM QAPP, the associated standard operating procedures [SOPs], and CAMs).

On November 1, 2017, Environmental Standards representative, Joseph P. Kraycik (the "Auditor"), conducted an audit of field activities associated with the weekly water-quality monitoring and sample collection performed by Anchor QEA personnel. The objective of the audited activities was to manually collect representative water samples from the Thompson Island, Lock 5, and Waterford sampling stations in the Hudson River.

The November 1, 2017 audit by Environmental Standards focused on an evaluation of several important aspects of the field activities being performed on that day. The following general headings were used to group field-audit activities for evaluation purposes:

1. Pre-Task Planning, Coordination, and Management
2. Field Documentation/Records
3. Supplies, Equipment, and Preparation
4. Field Sampling Procedures
5. Decontamination and Waste Management Procedures
6. Quality Assurance/Quality Control Sample Collection
7. Sample Packaging and Shipping
8. Sample Custody
9. Training and Health & Safety
10. General Observations

During the field audit, the Auditor utilized a field audit checklist that was developed specifically for the Water Monitoring Program field activities. This checklist contained pertinent items under each of the previously cited headings and provided the basic guidelines for a comprehensive audit.

Due to the nature of a field audit, some critical statements are presented in this report. These statements are based on observations made in the field and address those areas in which project Field Team deficiencies were noted and/or where changes may be appropriate. An exhaustive list of those activities performed in accordance with the project control documents and observed to be in compliance with standard industry protocol is not presented in this audit report; rather, this report identifies the type and source of deviations from the primary project control document (Phase 2 RAM QAPP, which contains the relevant SOPs for the project) as well as industry standards/best management practices.

2.0 Field Activities

The following section describes the field activities conducted during the November 1, 2017 field audit. The Auditor accompanied the Anchor QEA Field Team during the entire water monitoring event.

2.1 Manual Water Sample Collection

The Auditor observed water sampling activities associated with the Off-Season Water Column Monitoring Program. Specifically, the Auditor observed collection of water samples at the Lock 5 (Schuylerville) and Waterford sampling stations along the Upper Hudson River.

Sampling and monitoring activities observed during the audit were conducted by Anchor QEA Field Team representatives Chris Yates and Polomba Spina. The monitoring event observed by the Auditor generally consisted of visiting each of the monitoring locations for the manual collection of composite water samples using a Multiple Aliquot Depth Integrating Sampler (MADIS). Prior to sample collection at each sampling location, a YSI 6920 Multiparameter Water Quality Sonde was lowered into the river to measure water-quality data (including dissolved oxygen, pH, temperature, turbidity, and conductivity).

The MADIS, equipped with pre-cleaned glass collection vessels, was used to collect water samples from multiple sampling nodes along a transect at each sampling station. The MADIS sampler was lowered at each node to approximately 1 to 2 feet above the river bottom and then raised through the water column. The water samples collected from each node were then composited into a single sample representing the station transect.

Water samples collected during the November 1, 2017 field audit for total suspended solids (TSS) analysis were transported later the same day by a Pace Analytical Services, Inc. (Pace) courier to the Pace service center in Schenectady, New York. According to the Field Team, the TSS samples would then be shipped to the Pace Minneapolis, Minnesota laboratory for analysis. The samples collected for PCB analysis were packaged by the Field Team for shipment via Federal Express to Vista Analytical Laboratory (Vista) for analysis. US EPA did not provide oversight during the sampling activities on November 1, 2017.

Field activities performed by Anchor QEA Field Team personnel (hereinafter referred to as the "Field Team") and observed by the Auditor during the performance of the Water Monitoring Program activities were consistent with those described above, except as otherwise noted.

3.0 Audit Findings

During the field activities, the Auditor observed the Field Team conduct water monitoring and sampling activities to evaluate field performance and compliance with the project control documents. Observations of field procedures were recorded on a field audit checklist. The following sections provide the findings of the field audit and follow the major headings presented in Section 1. Comments made are followed by recommendations that were presented to the Field Team for consideration during the implementation of the project scope and when conducting future sampling programs and related field activities.

3.1 Pre-Task Planning, Coordination, and Management

The Auditor verified that the Field Team conducted daily pre-task planning meetings that addressed topics such as the project scope and objectives, identification of personnel and responsibilities, and health and safety issues. The Field Team debriefed the Auditor on field activity-specific topics and health and safety protocol prior to the start of field activities.

The Auditor verified that the Field Team was familiar with the project control documents, objectives, and scope prior to implementation of field activities. Copies of the project control documents were generally kept on-hand by the Field Team and were available for reference on field laptop computers used during monitoring activities. The Field Team was observed to be generally well prepared for the field activities and had adequately planned and coordinated activities with the appropriate project personnel.

No deficiencies associated with pre-task planning, coordination, and management were observed or reported.

3.2 Field Documentation and Records

Field documentation maintained by the Field Team primarily consisted of individual sample-specific electronic logs that were completed on a laptop computer and uploaded into the project electronic database. The electronic logs were used to record specific field measurements, data, and pertinent observations associated with the investigative samples. Individual sample labels and collective sample Chain-of-Custody (COC) forms were generated from this database. The Auditor verified that documentation of field data in the electronic database was complete, accurate, and consistent with Phase 2 RAM QAPP requirements.

A secondary form of field documentation occasionally maintained by the Field Team included a bound, dedicated project field logbook and project-specific forms. When in use, the logbook was utilized to record brief details or notes pertaining to daily field activities. Although the Phase 2 RAM QAPP does not specifically require the use of a logbook for field documentation purposes, the use of such a record is beneficial to the project because a field logbook provides a standalone record of pertinent data and details generated during field activities and also allows for the documentation of relevant information that cannot be stored in the project database under the current format. Use of a logbook for field documentation purposes is a standard industry practice. In addition, a YSI Calibration Log form was used to document calibration of the YSI Multiparameter Water Quality Sonde, which was conducted by Mr. Yates prior to sampling.

No deficiencies associated with field documentation and records were observed or reported.

3.3 Supplies, Equipment, and Preparation

The Auditor verified that field equipment and sampling supplies available for use during the field activities were consistent with the requirements of the Phase 2 RAM QAPP. The Field Team reported to the Auditor that field equipment inspection, calibration, and maintenance activities and maintenance of logs documenting these activities were performed by trained personnel in accordance with the frequencies required in the Phase 2 RAM QAPP. Calibration of YSI multi-parameter meter was documented on an equipment calibration form; these forms were stored in a 3-ring binder.

The Auditor verified that a sufficient number of certified-clean sample containers (consistent with the requirements of the Phase 2 RAM QAPP), sample coolers, custody seals, and other related materials had been obtained from the analytical laboratory to execute the sampling program.

No deficiencies associated with supplies, equipment, and preparation were observed or reported.

3.4 Field Sampling Procedures

The Auditor observed the field sampling procedures performed by the Field Team during the water monitoring event on November 1, 2017. The collection and handling of water-quality data and water samples (as discussed in the Field Activities section of this report) were performed in a consistent manner during the observed monitoring event. Field sampling activities were generally conducted in accordance with project control document requirements and industry standards.

No deficiencies associated with field sampling procedures were observed or reported.

3.5 Decontamination and Waste Management Procedures

The MADIS glass sample collection vessels were decontaminated by Pace after each use. The vessels were stored in racks and covered with aluminum foil until they were ready for use. The vessels were used one time and then returned to Pace for decontamination. The MADIS collection vessel caps and nozzles were dedicated to each sampling station and were rinsed with deionized water after use. Between uses, the caps and nozzles were stored in dedicated plastic containers that were labeled with the station name.

During monitoring activities, excess water produced from sampling activities was discharged to the Hudson River; disposable materials used during sampling activities were bagged for proper disposal.

No deficiencies associated with decontamination and waste management procedures were observed or reported.

3.6 Quality Assurance/Quality Control Sample Collection

The Auditor observed the collection of quality assurance/quality control (QA/QC) samples during the field audit. Specifically, on November 1, 2017, the following QA/QC sample was collected:

- A laboratory duplicate (LD) sample for TSS was collected at the Lock 5 (Schuylerville) location

No deficiencies associated with QA/QC sample collection were observed or reported.

3.7 Sample Packaging and Shipping

The Auditor observed sample packaging and shipping activities conducted by the Field Team during the monitoring event. Following collection, sample containers were placed in sealable plastic bags and were then placed in a cooler containing bagged wet ice. A temperature blank was placed in the cooler at the start of the sampling event. The Auditor also observed final packaging of the samples for relinquishment to the Pace laboratory courier and shipment via Federal Express to Vista. Samples were packaged in bubble wrap and additional cushioning material was added to each cooler to avoid breakage. Following packaging, the coolers were sealed with strapping tape and signed custody seals were applied to the coolers.

No deficiencies associated with sample packaging or shipping were observed or reported.

3.8 Sample Custody

The Auditor observed the Field Team complete electronic logs for individual samples at the time of collection and upload the sample-specific field data into the project electronic database. At the conclusion of sampling activities, COC documentation was generated by the database and maintained with the samples through delivery to the analytical laboratory. COC documentation contained complete and relevant information and was completed in accordance with Phase 2 RAM QAPP requirements. The COC Record for Pace was handed to the courier at the time of transfer; whereas, the COC Record for Vista was placed inside a sealable plastic bag and was taped to the inside lid of the cooler.

No deficiencies associated with sample custody were observed or reported.

3.9 Training and Health & Safety

The Field Team reported to the Auditor that each Field Team member had successfully completed the required project health and safety training and project task-specific training as necessary, and that each Field Team member possessed current Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) training certifications under 29 CFR 1910.120.

The Auditor verified that the Field Team was familiar with the project health and safety plan (HASP) and that a copy of the HASP was available for reference during field activities. Health and safety issues were addressed during a pre-task meeting between the Auditor and Field Team (discussed in Section 3.1 of this report). The Auditor observed that general health and safety procedures were followed during the field audit and that proper personal protective equipment (PPE) was available and in-use by the Field Team.

No deficiencies were observed or reported.

3.10 General Observations

The Field Team appeared to be well trained and was very familiar with the protocol for sample collection. Field Team personnel were organized, conducted field activities in a professional manner, and were receptive to recommendations made by the Auditor.

3.11 2017 Early Season Audit Findings

One audit finding was identified during the June 15, 2017 field audit:

- A review of the YSI 6920 Calibration Field Log indicated that the 40 NTU turbidity calibration standard was expired.

Subsequent to the field audit, the Field Team replaced the expired calibration standard with a new, unexpired standard and provided documentation to the Auditor. The Auditor confirmed that the calibration standards used during the November 1, 2017 field audit were within expiration dates.

4.0 Conclusions

The field audit performed by Environmental Standards identified that the Anchor QEA Field Team conducted its work in a professional manner and complied with the procedures described in the Phase 2 RAM QAPP and associated SOPs.

The Field Team executed the field procedures effectively and expressed a positive attitude about QA considerations for the project. Based on the Auditor's observations, the overall quality of services performed by the Field Team was satisfactory.

Audit Performed by,

Joseph P. Kraycik, P.G., CQA
Consulting Geoscientist

Report Reviewed by,

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Principal Chemist

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Date: November 28, 2017

FIELD AUDIT REPORT

OFF-SEASON WATER MONITORING –

PERFORMED ON JUNE 15, 2017

HUDSON RIVER PCBs SITE

2017 REMEDIAL ACTION MONITORING PROGRAM

July 12, 2017

Prepared for:

GENERAL ELECTRIC COMPANY
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Prepared by:

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Executive Summary

The field audit performed by Environmental Standards, Inc., identified that the Anchor QEA, LLC, Field Team conducted its work in a professional manner and complied with the majority of the procedures described in the Phase 2 Remedial Action Monitoring Quality Assurance Project Plan and associated standard operating procedures. One audit finding was identified (addressed in Section 3.0 of this Audit Report) and is summarized below:

- Field Sampling Procedures
    - A review of the YSI 6920 Calibration Field Log indicated that the 40 NTU turbidity calibration standard was expired.

Subsequent to the field audit, the Field Team replaced the expired calibration standard with a new, unexpired standard and provided documentation to the Auditor.

The deficiency identified in this audit report was generally minor in nature, was corrected in a timely manner, and likely did not result in a major impact to sample quality or jeopardize the data quality objectives of the project.

The Field Team executed the field procedures effectively and expressed a positive attitude about quality assurance considerations for the project. Based on the Auditor's observations, the overall quality of services performed by the Field Team was satisfactory.

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1.0 Introduction

The purpose and background for the remediation of the Hudson River Polychlorinated Biphenyls (PCBs) Site are presented in the Record of Decision (ROD) issued by the United States Environmental Protection Agency (US EPA) in February 2002. The ROD provided for the dredging of specified sediments in the Upper Hudson River that meet certain criteria, as well as the establishment of certain performance standards to govern implementation of the remedial action. The ROD stipulated the performance of this remedial action in two phases. The first phase (Phase 1) was the first season of dredging operations, during which, dredging occurred at a reduced rate and extensive monitoring was conducted. Phase 1 was conducted in the Spring, Summer, and Fall of 2009. The second phase of the remedial action (Phase 2), which began in 2011, consists of the remainder of the necessary dredging. Dredging was completed in 2015.

The Phase 2 Remedial Action Monitoring Program (RAMP) for 2017 generally includes the following monitoring components:

- Collection and analysis of water and fish samples for the purpose of determining the PCB concentrations that exist during remediation in the region of the Hudson River subject to remediation and in the broader region expected to benefit from the selected remedy.
- Monitoring of the suspended solids levels in water as an indicator of project-related sediment releases and monitoring of the levels of dissolved oxygen (DO) and pH in water to ascertain whether remediation is causing changes that could be harmful to aquatic biota or exceed drinking water standards.

RAMP activities conducted during the performance of Phase 2 of the remedial action are governed by the Hudson River PCBs Superfund Site Phase 2 Remedial Action Monitoring Quality Assurance Project Plan (Phase 2 RAM QAPP, May 2012). The Phase 2 RAM QAPP addresses collection, analysis, and data management activities of samples collected in 2017 as part of Phase 2 of the remedial action. A revised version of Attachment A to the RAM QAPP is currently being reviewed by US EPA. Corrective Action Memos (CAMs) are issued, as needed, to modify or amend the Phase 2 RAM QAPP. A Long-Term Operation, Maintenance, and Monitoring Plan for Water, Fish, and Sediment Monitoring (OMM Plan) is currently being prepared for the project and, upon approval, will supersede the Phase 2 RAM QAPP.

The General Electric Company (GE) retained Environmental Standards, Inc. (Environmental Standards), to conduct audits of specific field activities associated with the implementation of the Phase 2 RAMP. Specifically, the subject of this Field Audit Report is the audit (performed by Environmental Standards) of sampling and monitoring activities associated with the following program:

- Off-Season Water Column Monitoring

The audited activities were conducted by GE's environmental contractor, Anchor QEA, LLC (Anchor QEA).

This audit was performed to evaluate and document the overall quality of field services provided by the contractor (Anchor QEA) and to verify conformance of field procedures with project

requirements as specified in the project control documents (*i.e*., Phase 2 RAM QAPP, the associated standard operating procedures [SOPs], and CAMs).

On June 15, 2017, Environmental Standards, Inc. representative, Joseph P. Kraycik (the "Auditor"), conducted an audit of field activities associated with the weekly water-quality monitoring and sample collection performed by Anchor QEA personnel. The objective of the audited activities was to manually collect representative water samples from the Thompson Island, Lock 5, and Waterford sampling stations in the Hudson River.

The June 15, 2017 audit by Environmental Standards focused on an evaluation of several important aspects of the field activities being performed on that day. The following general headings were used to group field-audit activities for evaluation purposes:

1. Pre-Task Planning, Coordination, and Management
2. Field Documentation/Records
3. Supplies, Equipment, and Preparation
4. Field Sampling Procedures
5. Decontamination and Waste Management Procedures
6. Quality Assurance/Quality Control Sample Collection
7. Sample Packaging and Shipping
8. Sample Custody
9. Training and Health & Safety
10. General Observations

During the field audit, the Auditor utilized a field audit checklist that was developed specifically for the Water Monitoring Program field activities. This checklist contained pertinent items under each of the previously cited headings and provided the basic guidelines for a comprehensive audit.

Due to the nature of a field audit, some critical statements are presented in this report. These statements are based on observations made in the field and address those areas in which project Field Team deficiencies were noted and/or where changes may be appropriate. An exhaustive list of those activities performed in accordance with the project control documents and observed to be in compliance with standard industry protocol is not presented in this audit report; rather, this report identifies the type and source of deviations from the primary project control document (Phase 2 RAM QAPP, which contains the relevant SOPs for the project) as well as industry standards/best management practices.

2.0 Field Activities

The following section describes the field activities conducted during the June 15, 2017, field audit. The Auditor accompanied the Anchor QEA Field Team during the entire water monitoring event.

2.1 Manual Water Sample Collection

The Auditor observed water sampling activities associated with the Off-Season Water Column Monitoring Program. Specifically, the Auditor observed collection of water samples at the Thompson Island, Lock 5 (Schuylerville), and Waterford sampling stations along the Upper Hudson River.

Sampling and monitoring activities observed during the audit were conducted by Anchor QEA Field Team representatives Chris Yates and Kevin Ballou. The monitoring event observed by the Auditor generally consisted of visiting each of the monitoring locations for the manual collection of composite water samples using a Multiple Aliquot Depth Integrating Sampler (MADIS). Prior to sample collection at each sampling location, a YSI 6920 Multiparameter Water Quality Sonde was lowered into the river to measure water-quality data (including dissolved oxygen, pH, temperature, turbidity, and conductivity).

The MADIS, equipped with pre-cleaned glass collection vessels, was used to collect water samples from multiple sampling nodes along a transect at each sampling station. The MADIS sampler was lowered at each node to approximately 1 to 2 feet above the river bottom and then raised through the water column. The water samples collected from each node were then composited into a single sample representing the station transect.

Water samples collected during the June 15, 2017 field audit were transported on June 16, 2017, by a Pace Analytical Services, Inc. (Pace) courier to the Pace service center in Schenectady, New York. According to the Field Team, samples for total suspended solids (TSS) analysis would be shipped to the Pace Minneapolis, Minnesota laboratory for analysis. The samples collected for PCB analysis would be held in cold storage at the Pace Schenectady service center until Vista Analytical Laboratory (Vista) is approved by US EPA, at which time, those samples would be shipped to Vista for analysis. US EPA did not provide oversight during the sampling activities on June 15, 2016.

Field activities performed by Anchor QEA Field Team personnel (hereinafter referred to as the "Field Team") and observed by the Auditor during the performance of the Water Monitoring Program activities were consistent with those described above, except as otherwise noted.

3.0 Audit Findings

During the field activities, the Auditor observed the Field Team conduct water monitoring and sampling activities to evaluate field performance and compliance with the project control documents. Observations of field procedures were recorded on a field audit checklist. The following sections provide the findings of the field audit and follow the major headings presented in Section 1. Comments made are followed by recommendations that were presented to the Field Team for consideration during the implementation of the project scope and when conducting future sampling programs and related field activities.

3.1 Pre-Task Planning, Coordination, and Management

The Auditor verified that the Field Team conducted daily pre-task planning meetings that addressed topics such as the project scope and objectives, identification of personnel and responsibilities, and health and safety issues. The Field Team debriefed the Auditor on field activity-specific topics and health and safety protocol prior to the start of field activities.

The Auditor verified that the Field Team was familiar with the project control documents, objectives, and scope prior to implementation of field activities. Copies of the project control documents were generally kept on-hand by the Field Team and were available for reference on field laptop computers used during monitoring activities. The Field Team was observed to be generally well prepared for the field activities and had adequately planned and coordinated activities with the appropriate project personnel.

No deficiencies associated with pre-task planning, coordination, and management were observed or reported.

3.2 Field Documentation and Records

Field documentation maintained by the Field Team primarily consisted of individual sample-specific electronic logs that were completed on a laptop computer and uploaded into the project electronic database. The electronic logs were used to record specific field measurements, data, and pertinent observations associated with the investigative samples. Individual sample labels and collective sample Chain-of-Custody (COC) forms were generated from this database. The Auditor verified that documentation of field data in the electronic database was complete, accurate, and consistent with Phase 2 RAM QAPP requirements.

A secondary form of field documentation occasionally maintained by the Field Team included a bound, dedicated project field logbook and project-specific forms. When in use, the logbook was utilized to record brief details or notes pertaining to daily field activities. Although the Phase 2 RAM QAPP does not specifically require the use of a logbook for field documentation purposes, the use of such a record is beneficial to the project because a field logbook provides a stand-alone record of pertinent data and details generated during field activities and also allows for the documentation of relevant information that cannot be stored in the project database under the current format. Use of a logbook for field documentation purposes is a standard industry practice. In addition, a YSI Calibration Log form was used to document calibration of the YSI Multiparameter Water Quality Sonde which was conducted by Mr. Yates prior to sampling.

No deficiencies associated with field documentation and records were observed or reported.

3.3 Supplies, Equipment, and Preparation

The Auditor verified that field equipment and sampling supplies available for use during the field activities were consistent with the requirements of the Phase 2 RAM QAPP. The Field Team reported to the Auditor that field equipment inspection, calibration, and maintenance activities and maintenance of logs documenting these activities were performed by trained personnel in accordance with the frequencies required in the Phase 2 RAM QAPP. Calibration of YSI multi-parameter meter was documented on an equipment calibration form; these forms were stored in a 3-ring binder.

The Auditor verified that a sufficient number of certified-clean sample containers (consistent with the requirements of the Phase 2 RAM QAPP), sample coolers, custody seals, and other related materials had been obtained from the analytical laboratory to initiate the sampling program.

Finding: During a review of the YSI Calibration Field Log dated June 15, 2017, the Auditor observed that the 40 NTU turbidity calibration standard expired in May 2017.

Recommendation: The Auditor recommended that the expired calibration standard be replaced with an unexpired standard. Subsequent to the field audit, the Field Team replaced the expired standard with a non-expired standard. The replacement standard was manufactured by AMCOClear with an expiration date of April 2018.

3.4 Field Sampling Procedures

The Auditor observed the field sampling procedures performed by the Field Team during the water monitoring event on June 15, 2017. The collection and handling of water-quality data and water samples (as discussed in the Field Activities section of this report) were performed in a consistent manner during the observed monitoring event. Field sampling activities were generally conducted in accordance with project control document requirements and industry standards.

No deficiencies associated with field sampling procedures were observed or reported.

3.5 Decontamination and Waste Management Procedures

The MADIS glass sample collection vessels are decontaminated by Pace after each use. The vessels are stored in racks and covered with aluminum foil until they are ready for use. The vessels are used one time and then returned to Pace for decontamination. The MADIS collection vessel caps and nozzles are dedicated to each sampling station and are rinsed with deionized water after use. Between uses, the caps and nozzles are stored in plastic containers that are labeled with the station name.

During monitoring activities, excess water produced from sampling activities was discharged to the Hudson River; disposable materials used during sampling activities were bagged for proper disposal.

No deficiencies associated with decontamination and waste management procedures were observed or reported.

3.6 Quality Assurance/Quality Control Sample Collection

The Auditor observed the collection of quality assurance/quality control (QA/QC) samples during the field audit. Specifically, on June 15, 2017, the following QA/QC sample was collected:

- A laboratory duplicate (LD) sample for TSS was collected at the Thompson Island location

No deficiencies associated with QA/QC sample collection were observed or reported.

3.7 Sample Packaging and Shipping

The Auditor observed sample packaging and shipping activities conducted by the Field Team during the monitoring event. Following collection, sample containers were placed in sealable plastic bags and were then placed in a cooler containing bagged wet ice. A temperature blank was placed in cooler at the start of the sampling event. The Auditor did not observe final packaging of the samples for relinquishment to the laboratory courier.

No deficiencies associated with sample packaging or shipping were observed or reported.

3.8 Sample Custody

The Auditor observed the Field Team complete electronic logs for individual samples at the time of collection and upload the sample-specific field data into the project electronic database. At the conclusion of sampling activities, COC documentation was generated by the database and maintained with the samples through delivery to the analytical laboratory. COC documentation contained complete and relevant information and was completed in accordance with Phase 2 RAM QAPP requirements.

No deficiencies associated with sample custody were observed or reported.

3.9 Training and Health & Safety

The Field Team reported to the Auditor that each Field Team member had successfully completed the required project health and safety training and project task-specific training as necessary, and that each Field Team member possessed current Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) training certifications under 29 CFR 1910.120.

The Auditor verified that the Field Team was familiar with the project health and safety plan (HASP) and that a copy of the HASP was available for reference during field activities. Health and safety issues were addressed during a pre-task meeting between the Auditor and Field Team (discussed in Section 3.1 of this report). The Auditor observed that general health and safety procedures were followed during the field audit and that proper personal protective equipment (PPE) was available and in-use by the Field Team.

No deficiencies were observed or reported.

3.10 General Observations

The Field Team appeared to be well trained and was very familiar with the protocol for sample collection. Field Team personnel were organized, conducted field activities in a professional manner, and were receptive to recommendations made by the Auditor.

3.11 2016 Audit Findings

Two audit findings were identified during the May 2016 field audit. Subsequent to the May 2016 field audit, the Project Team agreed that procedural modifications associated with the findings were warranted and issued a corrective action memo (CAM #13) to formally document these changes. The Field Team conducted field work in accordance with CAM #13 during the June 15, 2017 field audit.

4.0 Conclusions

The field audit performed by Environmental Standards identified that the Anchor QEA Field Team conducted its work in a professional manner and complied with the majority of the procedures described in the Phase 2 RAM QAPP and associated SOPs. One audit finding (addressed in Section 3.0) was identified and is summarized below:

- Field Sampling Procedures
    - A review of the YSI 6920 Calibration Field Log indicated that the 40 NTU turbidity calibration standard was expired.

Subsequent to the field audit, the Field Team replaced the expired calibration standard with a new, unexpired standard and provided documentation to the Auditor.

The deficiency identified in this audit report was generally minor in nature, was corrected in a timely manner, and likely did not result in a major impact to sample quality or jeopardize the data quality objectives of the project.

The Field Team executed the field procedures effectively and expressed a positive attitude about QA considerations for the project. Based on the Auditor's observations, the overall quality of services performed by the Field Team was satisfactory.

Audit Performed by,

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Report Reviewed by,

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Date: July 12, 2017

Appendix C
2017 Water Database

(Provided on accompanying DVD)

Appendix D
2017 Fish Database

(Provided on accompanying DVD)